PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 23 August 2001 (23.08.01)	
International application No. PCT/US00/29854	**Applicant's or agent's file reference** 310048-488WO
International filing date (day/month/year) 30 October 2000 (30.10.00)	**Priority date** (day/month/year) 29 October 1999 (29.10.99)
Applicant AKHAVE, Jay, R. et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

29 May 2001 (29.05.01)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland**	Authorized officer Olivia TEFY
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TRE TY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

To:

ROSE, Alan, C.
Oppenheimer Wolff & Donnelly LLP
2029 Century Park East
Suite 3800
Los Angeles, CA 90067
ETATS-UNIS D'AMERIQUE

Date of mailing (day/month/year)
08 November 2001 (08.11.01)

Applicant's or agent's file reference
310048-488WO

IMPORTANT NOTIFICATION

International application No.
PCT/US00/29854

International filing date (day/month/year)
30 October 2000 (30.10.00)

1. The following indications appeared on record concerning:
[X] the applicant [X] the inventor [ ] the agent [ ] the common representative

Name and Address

State of Nationality | State of Residence

Telephone No.

Facsimile No.

Teleprinter No.

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:
[X] the person [ ] the name [ ] the address [ ] the nationality [ ] the residence

Name and Address
LICON, Michael
226 North Rock River Drive
Diamond Br, CA 91765
United States of America

State of Nationality: US | State of Residence: US

Telephone No.

Facsimile No.

Teleprinter No.

3. Further observations, if necessary:
Addition of inventor and applicant for US only.

4. A copy of this notification has been sent to:
[X] the receiving Office
[ ] the International Searching Authority
[X] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

Authorized officer
Anman QIU
Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

To:

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08 November 2001 (08.11.01)

Applicant's or agent's file reference
310048-488WO

IMPORTANT NOTIFICATION

International application No.
PCT/US00/29854

International filing date (day/month/year)
30 October 2000 (30.10.00)

1. The following indications appeared on record concerning:

[X] the applicant [X] the inventor [ ] the agent [ ] the common representative

Name and Address	State of Nationality	State of Residence
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[X] the person [ ] the name [ ] the address [ ] the nationality [ ] the residence

Name and Address	State of Nationality	State of Residence
LICON, Michael 226 North Rock River Drive Diamond Br, CA 91765 United States of America	US	US
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:
Addition of inventor and applicant for US only.

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[X] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Anman QIU
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ US

PCT

CHAPTER II

DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty and hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only

Identification of IPEA	Date of receipt of DEMAND

Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION

Applicant's or agent's file reference: 310048-488WO

International application No.	International filing date *(day/month/year)*	(Earliest) Priority date *(day/month/year)*
PCT/US/OO/29854	30.October.2000 (30.10.00)	29.October.99 (29.10.99)

Title of invention:
AN APPARATUS FOR HIGH-THROUGHPUT PRODUCTION OF COAT MATERIAL ARRAYS, AND ANALYTICAL METHODS USING SUCH ARRAYS

Box No. II APPLICANT(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*
(applicant, all designated states except the U.S.)
Avery Dennison Corporation
150 North Orange Grove Boulevard
Pasadena, California 91103
United States of America

Telephone No.: --
Facsimile No.: --
Teleprinter No.: --
Applicant's registration No. with the Office: --

State *(that is, country)* of nationality:	State *(that is, country)* of residence:
US	US

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*
(applicant and inventor)(U.S. only)
AKHAVE, Jay, R.
845 Pomello Drive
Claremont, California 91711
United States of America

State *(that is, country)* of nationality:	State *(that is, country)* of residence:
US	US

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*
(applicant and inventor)(U.S. only)
SAUNDERS, Dennis, L.
2059 Paseo Ambar
San Dimas, California 91773
United States of America

State *(that is, country)* of nationality:	State *(that is, country)* of residence:
US	US

☐ Further applicants are indicated on a continuation sheet.

EL585709009US

Sheet No. 2

International application No.
PCT/US/OO/29854

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The following person is [X] agent [ ] common representative

and [ ] has been appointed earlier and represents the applicant(s) also for international preliminary examination.

[ ] is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.

[ ] is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

ROSE, Alan C.
OPPENHEIMER, WOLFF & DONNELLY LLP
2029 Century Park East, Suite 3800
Los Angeles, California 90067-3024
United States of America

Telephone No.
310.788.5030

Facsimile No.
310.788.5100

Teleprinter No.
arose@oppenheimer.com

Agent's registration No. with the Office
17,047

[ ] **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION

Statement concerning amendments:*

1. The applicant wishes the international preliminary examination **to start on the basis of:**

[X] the international application as originally filed

the description [X] as originally filed
[ ] as amended under Article 34

the claims [X] as originally filed
[ ] as amended under Article 19 (together with any accompanying statement)
[ ] as amended under Article 34

the drawings [X] as originally filed
[ ] as amended under Article 34

with all responses to Invitations having been considered.

2. [ ] The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.

3. [ ] The applicant wishes the start of the international preliminary examination **to be postponed** until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination: ENGLISH

[X] which is the language in which the international application was filed.
[ ] which is the language of a translation furnished for the purposes of international search.
[ ] which is the language of publication of the international application.
[ ] which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

Box No. V ELECTION OF STATES

The applicant hereby **elects all eligible States** *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*

excluding the following States which the applicant wishes **not to elect:**

Sheet No. . . .

International application No.
PCT/US/OO/29854

Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

			For International Preliminary Examining Authority use only: received	not received
1. translation of international application	:	sheets	☐	☐
2. amendments under Article 34	:	sheets	☐	☐
3. copy (or, where required, translation) of amendments under Article 19	:	sheets	☐	☐
4. copy (or, where required, translation) of statement under Article 19	:	sheets	☐	☐
5. letter	:	sheets	☐	☐
6. other *(specify)*	:	sheets	☐	☐

The demand is also accompanied by the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ original separate power of attorney
3. ☐ original general power of attorney
4. ☐ copy of general power of attorney; reference number, if any:
5. ☐ statement explaining lack of signature
6. ☐ sequence listing in computer readable form
7. ☐ other *(specify)*:

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).

Alan C. Rose

Alan C. Rose, Esq.
OPPENHEIMER WOLFF & DONNELLY LLP

For International Preliminary Examining Authority use only

1. Date of actual receipt of DEMAND:
2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):
3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply. ☐ The applicant has been informed accordingly.
4. ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.
5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

Demand received from IPEA on:

CHAPTER II

PCT

FEE CALCULATION SHEET

Annex to the Demand

International application No. PCT/US/OO/29854	For International Preliminary Examining Authority use only
Applicant's or agent's file reference 310048-488WO	Date stamp of the IPEA

Applicant
Avery Dennison Corporation

CALCULATION OF PRESCRIBED FEES

1. Preliminary examination fee	490.00 P	
2. Handling fee *(Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.)*	137.00 H	
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box	527.00 TOTAL	

MODE OF PAYMENT

- ☐ authorization to charge deposit account with the IPEA (see below)
- ☒ cheque
- ☐ postal money order
- ☐ bank draft
- ☐ cash
- ☐ revenue stamps
- ☐ coupons
- ☐ other *(specify):*

AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT

(This mode of payment may not be available at all IPEAs)

IPEA/ US

☐ Authorization to charge the total fees indicated above.

☒ *(This check-box may be marked only if the conditions for deposit accounts of the IPEA so permit)* Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.

Deposit Account No.: 16-22230

Date: 29.May.2001

Name: Alan C. Rose, Esq.

Signature: Alan C. Rose

See Notes to the fee calculation sheet

PATENT COOPERATION TREATY

PCT

From the INTERNATIONAL BUREAU

To:
ROSE, Alan, C.
Oppenheimer Wolff & Donnelly LLP
2029 Century Park East
Suite 3800
Los Angeles, CA 90067
ETATS-UNIS D'AMERIQUE

NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

Date of mailing (day/month/year) 10 May 2001 (10.05.01)		
Applicant's or agent's file reference 310048-488WO		**IMPORTANT NOTICE**
International application No. PCT/US00/29854	**International filing date** (day/month/year) 30 October 2000 (30.10.00)	**Priority date** (day/month/year) 29 October 1999 (29.10.99)
Applicant AVERY DENNISON CORPORATION et al		

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:
   AU,KP,KR,US

   In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:
   AE,AG,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,BZ,CA,CH,CN,CR,CU,CZ,DE,DK,DM,DZ,EA,EE,EP,ES, FI,GB,GD,GE,GH,GM,HR,HU,ID,IL,IN,IS,JP,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,MG,MK, MN,MW,MX,MZ,NO,NZ,OA,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,UZ,VN,YU,
   The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on
   10 May 2001 (10.05.01) under No. WO 01/33211

REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, **a demand for international preliminary examination** must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the **national phase**, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No. (41-22) 740.14.35

Authorized officer
J. Zahra
Telephone No. (41-22) 338.83.38

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To: ALAN C. ROSE
OPPENHEIMER WOLFF & DONNELLY LLP
2029 CENTURY PARK EAST, SUITE 3800
LOS ANGELES, CA 90067

PCT

WRITTEN OPINION

(PCT Rule 66)

	Date of Mailing *(day/month/year)*
Applicant's or agent's file reference 310048-488WO	**REPLY DUE** within ONE months from the above date of mailing

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/US00/29854	30 OCTOBER 2000	29 OCTOBER 1999

International Patent Classification (IPC) or both national classification and IPC
See Supplemental Sheet.

..ERY DENNISON CORPORATION

1. This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.

2. This opinion contains indications relating to the following items:

- I [X] Basis of the opinion
- II [ ] Priority
- III [ ] Non-establishment of opinion with regard to novelty, inventive step or industrial applicability
- IV [ ] Lack of unity of invention
- V [X] Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI [ ] Certain documents cited
- VII [ ] Certain defects in the international application
- VIII [ ] Certain observations on the international application

3. The applicant is hereby invited to reply to this opinion.

When? See the time limit indicated above. ~~The applicant may, before the expiration of that time limit, request this Authority to grant an extension., see Rule 66.2(d).~~

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 *bis*.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.

4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 28 FEBRUARY 2002

Name and mailing address of the IPEA/US	Authorized officer
Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	ARLEN SODERQUIST Jean Proctor Paralegal Specialist
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0661

Form PCT/IPEA/408 (cover sheet) (July 1998)★

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To: ALAN C. ROSE
OPPENHEIMER WOLFF & DONNELLY LLP
2029 CENTURY PARK EAST, SUITE 3800
LOS ANGELES, CA 90067

WRITTEN OPINION

(PCT Rule 66)

Date of Mailing *(day/month/year)*	17 SEP 2001
Applicant's or agent's file reference: 310048-488WO	REPLY DUE within ONE months from the above date of mailing

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/US00/29854	30 OCTOBER 2000	29 OCTOBER 1999

International Patent Classification (IPC) or both national classification and IPC
Please See Supplemental Sheet.

Applicant
AVERY DENNISON CORPORATION

1. This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.

2. This opinion contains indications relating to the following items:

- I [X] Basis of the opinion
- II [ ] Priority
- III [ ] Non-establishment of opinion with regard to novelty, inventive step or industrial applicability
- IV [ ] Lack of unity of invention
- V [X] Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI [ ] Certain documents cited
- VII [ ] Certain defects in the international application
- VIII [ ] Certain observations on the international application

3. The applicant is hereby invited to reply to this opinion.

When? See the time limit indicated above. ~~The applicant may, before the expiration of that time limit, request this Authority to grant an extension., see Rule 66.2(d).~~

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66.3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4.
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 *bis*.
For an informal communication with the examiner, see Rule 66.6.

If no reply is filed, the international preliminary examination report will be established on the basis of this opinion.

4. The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 28 FEBRUARY 2002

Name and mailing address of the IPEA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer
ARLEN SODERQUIST
Jean Proctor
Paralegal Specialist

Telephone No. (703) 308-0661

W[redacted]EN OPINION

International application No.
[redacted]CT/US00/29854

I. Basis of the opinion

1. With regard to the **elements** of the international application: *

[X] the international application as originally filed

[X] the description:
pages 1-12 , as originally filed
pages NONE , filed with the demand
pages NONE , filed with the letter of _____

[X] the claims:
pages 13-16 , as originally filed
pages NONE , as amended (together with any statement) under Article 19
pages NONE , filed with the demand
pages NONE , filed with the letter of _____

[X] the drawings:
pages 1-7 , as originally filed
pages NONE , filed with the demand
pages NONE , filed with the letter of _____

[X] the sequence listing part of the description:
pages NONE , as originally filed
pages NONE , filed with the demand
pages NONE , filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

[ ] the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

[ ] the language of publication of the international application (under Rule 48.3(b)).

[ ] the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

[ ] contained in the international application in printed form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readable form.

[ ] The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] The statement that the information recorded in computer readable form is identical to the writen sequence listing has been furnished.

4. [X] The amendments have resulted in the cancellation of:

[X] the description, pages NONE

[X] the claims, Nos. NONE

[X] the drawings, sheets/~~fig~~ NON

5. [ ] This opinion has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

** Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed".*

WRITTEN OPINION

International application No.
PCT/US00/29854

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. statement

Novelty (N)	Claims	1-9,12-20	YES
	Claims	10-11	NO
Inventive Step (IS)	Claims	NONE	YES
	Claims	1-20	NO
Industrial Applicability (IA)	Claims	1-20	YES
	Claims	NONE	NO

2. citations and explanations

Claims 10-11 lack novelty under PCT Article 33(2) as being anticipated by Peters. In the patent Peters teaches a receptacle for cell cultures or biological tests comprising a base plate (3), and a wall member (1) joined in detachable and liquid-tight manner to the base plate, the base plate and wall defining at least one chamber (2), at least that portion of the wall adjacent the base plate consisting of a noncytotoxic elastomeric synthetic material adhering to the base plate. Column 2, lines 8-36 teach the various materials for the two sections. It is noted that both preferred materials are flexible (see lines 29-34). The detachability facilitates the handling of the respective parts in use. The device is used to form and hold a plurality of test substances in defined regions on the base plate.

Claims 1-9 and 12-20 lack an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of Machevskaya et al. Peters does not teach using the device to test coatings.

In the paper Machevskaya et al. presents a study of the interrelation of properties of coatings and the composition of epoxy-phenol compositions. The solvent composition, Al_2O_3 content, and viscosity of coating compositions containing constant amounts of epoxy and phenol-formaldehyde resins and pigmented with iron oxide were optimized by 2^5 experimental design with respect to the thickness (h), thickness variation (h'), roughness (R), and abrasion resistance (V) of coatings applied by the centrifugal method. Analysis of the regression equations obtained showed that h decreased with increasing xylene content, h' increased with increasing cyclohexanone content and with increasing viscosity, R decreased with increasing viscosity because of the increase of centrifuge rotation velocity, and V increased with increasing Al_2O_3 content (3-6 weight% was the optimal value) and decreasing xylene content.

It would have been obvious to use the device of Peters for making the various compositions of Machevskaya et al. because of its recognized ability to form and hold a plurality of test substances in defined regions on a base plate followed by

(Continued on Supplemental Sheet.)

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

TIME LIMIT:

The time limit set for response to a Written Opinion may not be extended. 37 CFR 1.484(d). Any response received after the expiration of the time limit set in the Written Opinion will not be considered in preparing the International Preliminary Examination Report.

CLASSIFICATION:

The International Patent Classification (IPC) and/or the National classification are as listed below:
IPC(7): G01N 31/00, 1/28; C12M 1/34 and US Cl.: 422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183

V. 2. REASONED STATEMENTS - CITATIONS AND EXPLANATIONS (Continued):
subsequent testing of the materials.

Claims 1-20 meet the criteria set out in PCT Article 33(4), because they clearly could be used for testing of coating materials.

---------- NEW CITATIONS ----------
R. A. Machevskaya et al, "Study of the Interrelation of Properties of Coatings and the Composition of Epoxy-Phenol Compositions" Lakokras. Mater. Ikh Primen. 1981, pages 35-36, see attached abstract.

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To: ALAN C. ROSE
OPPENHEIMER WOLFF & DONNELLY LLP
2029 CENTURY PARK EAST, SUITE 3800
LOS ANGELES, CA 90067

PCT

NOTIFICATION OF TRANSMITTAL OF INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of Mailing (day/month/year)	22 JAN 2002
Applicant's or agent's file reference: 310048-488WO	IMPORTANT NOTIFICATION

International application No.	International filing date (day/month/year)	Priority Date (day/month/year)
PCT/US00/29854	30 OCTOBER 2000	29 OCTOBER 1999

Applicant
AVERY DENNISON CORPORATION

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer ARLEN SODERQUIST
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0661

Form PCT/IPEA/416 (July 1992)★

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 310048-488WO	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/US00/29854	International filing date (day/month/year) 30 OCTOBER 2000	Priority date (day/month/year) 29 OCTOBER 1999
International Patent Classification (IPC) or national classification and IPC Please See Supplemental Sheet.		
Applicant AVERY DENNISON CORPORATION		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 34 sheets.

   [✓] This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 3 sheets.

3. This report contains indications relating to the following items:

   I [X] Basis of the report

   II [ ] Priority

   III [ ] Non-establishment of report with regard to novelty, inventive step or industrial applicability

   IV [ ] Lack of unity of invention

   V [X] Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

   VI [ ] Certain documents cited

   VII [ ] Certain defects in the international application

   VIII [ ] Certain observations on the international application

Date of submission of the demand	Date of completion of this report
29 MAY 2001	12 DECEMBER 2001
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer ARLEN SODERQUIST Telephone No. (703) 308-0661

Form PCT/IPEA/409 (cover sheet) (July 1998)*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US00/29854

I. Basis of the report

1. With regard to the **elements** of the international application:*

☐ the international application as originally filed

☒ the description:
pages (See Attached), as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____

☒ the claims:
pages (See Attached), as originally filed
pages _____, as amended (together with any statement) under Article 19
pages _____, filed with the demand
pages _____, filed with the letter of _____

☒ the drawings:
pages (See Attached), as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____

☒ the sequence listing part of the description:
pages (See Attached), as originally filed
pages _____, filed with the demand
pages _____, filed with the letter of _____

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).

☐ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in printed form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the writen sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

☒ the description, pages NONE

☒ the claims, Nos. 19-20

☒ the drawings, sheets/~~fig~~ NONE

5. ☐ This report has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

** Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

***Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US00/29854

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. statement

Novelty (N)	Claims	1-18	YES
	Claims	NONE	NO
Inventive Step (IS)	Claims	1-18	YES
	Claims	NONE	NO
Industrial Applicability (IA)	Claims	1-18	YES
	Claims	NONE	NO

2. citations and explanations (Rule 70.7)

Claims 1-18 meet the criteria set out in PCT Article 33(2)-(4), because the prior art does not teach or fairly suggest A method as claimed in which a centrifuge is used to form a plurality of sample coatings using the claimed substrate as found in claim 1.

R. A. Machevskaya et al, "Study of the Interrelation of Properties of Coatings and the Composition of Epoxy-Phenol Compositions" Lakokras. Mater. Ikh Primen. 1981, pages 35-36, see attached abstract.

Supplemental Box
(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

CLASSIFICATION:
The International Patent Classification (IPC) and/or the National classification are as listed below:
IPC(7): G01N 31/00, 1/28; C12M 1/34 and US Cl.: 422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183

I. BASIS OF REPORT:

This report has been drawn on the basis of the description,
page(s) 1-12, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:
NONE

This report has been drawn on the basis of the claims,
page(s) 13, as originally filed.
page(s) NONE, as amended under Article 19.
page(s) NONE, filed with the demand.
and additional amendments:
Claim pages 14-16, filed with the letter of 16 October 2001.

This report has been drawn on the basis of the drawings,
page(s) 1-7, as originally filed.
page(s) NONE, filed with the demand.
and additional amendments:
NONE

This report has been drawn on the basis of the sequence listing part of the description:
page(s) NONE, as originally filed.
pages(s) NONE, filed with the demand.
and additional amendments:
NONE

mounting said receptacle in a centrifuge with the outward centrifugal force being perpendicular to the bottom of said receptacle;

activating said centrifuge to flatten the material in the receptacle; and

drying said material while the sample is being rotated and flattened by the centrifugal action.

8. A method for efficiently preparing a large number of sample castings comprising the steps of:

forming a series of sample receptacles by providing a substrate and an overlying apertured sheet with the apertured sheet in tight sealing engagement with the substrate;

applying different samples of material in liquid form into said receptacles;

drying said samples; and

removing said apertured sheet to leave said material samples on said substrate.

9. A method as defined in claim 8 including the step of applying force to said samples perpendicular to the bottom of said receptacles to flatten out said samples.

10. A method of testing coating materials, comprising the steps of:

providing an array of coating wells, each well being configured for receiving a coating material having a known composition;

placing a coating material having a known composition in each coating well, varying the composition so as to provide a plurality of coating materials having different compositions in a plurality of coating wells;

correlating the composition of the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a specific composition is associated with each coating well position in the array;

placing said coating wells with said compositions into a centrifuge, and activating said centrifuge;

drying said coating materials; and

testing the resultant coatings.

11. The method of claim 10 including providing wells in the form of a flexible substitute and a flexible overlying apertured sheet.

12. The method of claim 10 including the step of heating said coating materials while said centrifuge is activated.

13. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter; said array of coating wells comprising a substrate and an overlying apertured sheet;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

drying said coating samples; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

14. The method of claim 13, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after drying, whereby the coating material array is carried by the substrate alone after separation.

15. The method of claim 13, wherein the well depth and volume is substantially greater than that of the coating volume.

16. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

applying a centrifugal force to the array of coating wells to level the coating material in the coating wells;

curing said coating samples under said coating leveling force; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

17. The method of claim 16, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after application of the leveling force, whereby the coating material array is carried by the substrate alone after separation.

18. The method of claim 10, further comprising the steps of:

curving the said array of coating wells to substantially match the curvature of the curvilinear path of the array during centrifuging.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

A. CLASSIFICATION OF SUBJECT MATTER
IPC(7) :G01N 31/00, 1/28; C12M 1/34
US CL :422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X - Y	US 4,299,920 A (PETERS) 10 November 1981, see entire document.	10-11 --- 1-9,12-20
Y	Chemical Abstracts, Vol. 94, No. 16, issued 20 April 1981, R. A. Machevskaya et al, "Study of the interrelation of Propeties of Coatings and the Composition of Epoxy-Phenol Compositions" see page 93, Col. 1, abstract No. 123179p, Lakokras. Mater. Ikh Primen., 1981, (1), 35-36.	1-20
Y,E	US 5,985,356 A (SCHULTZ et al) 16 November 1999, see entire document.	1-20
A	CA 2,260,807 A (EIPEL et al) 29 January 1998.	1-20

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
18 JANUARY 2001	31 JAN 2001
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer ARLEN SODERQUIST Jean Proctor Paralegal Specialist Telephone No. (703) 308-0661

Form PCT/ISA/210 (second sheet) (July 1998)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 363,504 (PPG HELLIGE B.V.) 18 April 1990.	1-20

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

B. FIELDS SEARCHED
Electronic data bases consulted (Name of data base and where practicable terms used):

STN search in CA file
search terms: coating or adhesive or protect?, layer, film, combinator?, library, centrifug?, dry dried drying, cure, curing, cured

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
10 May 2001 (10.05.2001)

PCT

(10) International Publication Number
WO 01/33211 A1

(51) International Patent Classification[7]: G01N 31/00, 1/28, C12M 1/34

(21) International Application Number: PCT/US00/29854

(22) International Filing Date: 30 October 2000 (30.10.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/162,349 29 October 1999 (29.10.1999) US

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(72) Inventors; and

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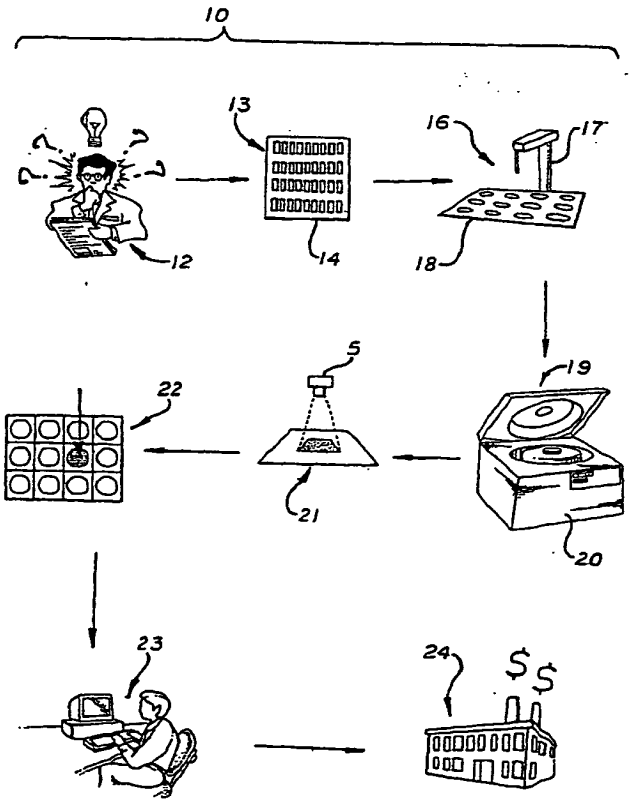
(74) Agents: ROSE, Alan, C. et al.; Oppenheimer Wolff & Donnelly LLP, 2029 Century Park East, Suite 3800, Los Angeles, CA 90067 (US).

(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,

[Continued on next page]

(54) Title: AN APPARATUS FOR HIGH-THROUGHPUT PRODUCTION OF COAT MATERIAL ARRAYS, AND ANALYTICAL METHODS USING SUCH ARRAYS



(57) Abstract: A combinatorial, high-throughput screening method is described for developing new coatings having a desired performance characteristic of a coating property which results in a substantial increase in the discovery rate of new coating materials. The method includes the steps of providing an array of wells (18) for receiving candidate coating materials having a known parameter; placing coating materials in each well (16) while varying the coating material parameter; correlating the coating material position in the array to the variation of the coating material parameter; applying a coating leveling force to and optionally drying the coating materials in the array of coating wells (19); testing the coatings with regard to the desired performance characteristic (21) and correlating the result of the test to the well position in the array that thereby coating materials having the desired performance characteristic may be discovered.

WO 01/33211 A1

IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

— *With international search report.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

An Apparatus for High-Thr ughput Production of Coat Material Arrays, and Analytical Methods Using such Arrays

Related Patent Application

This application claims priority from prior U. S. Provisional Patent Application Serial Number 60/162.349 filed October 29. 1999. the disclosure of which is hereby incorporated by reference.

Background:

1. Field of the invention:.

The invention relates generally to methods and apparatus for identification and optimization of coating materials and properties for desired applications. More specifically, the invention rclates to an improved process of creating coatings, involving identifying candidate materials and screening and optimizing formulations and coating parameters for desired applications.

2. Description of related art

Development of coating materials. for example adhesive coatings, release coats, protective coatings. and the like as well as films and laminate constructions of layered materials. has conventionally been a time consuming and labor intensive process. Candidate materials are identified primarily based on knowledge and experience with what compositions have worked before in related applications and investigating like materials and combinations of materials. This usually involves preparing a coating formulation. preparing a test coating for evaluation (often involving several tries to attain the desired parameters such as coat weight. cure. etc. for evaluation). drying the coating. then evaluating the coating by testing the property of interest. such as permeability. tack. shear and bending strength. surface roughness. etc.. and entering the results in a database for comparison with further coatings to be developed and tested. Problems of cross-contamination and holdover further limit the number of formulations that can be screened in a given time period. This is a time-consuming process and as a result one skilled in the art. even with support staff to assist and carry on tasks in parallel. has conventionally been able to screen at most a few coatings per day, most often only one or two.

Because of the lengthy time required to screen and then investigate candidate materials and associated coating application parameter values to select and optimize coatings. those skilled in the art generally must focus on families of materials known to possess properties likely to prove successful in the intended use. Investigation of unconventional or simply previously

untried materials is usually limited. Moreover, development of coating materials for a particular application is also a time-consuming process, and development of new coatings, while potentially beneficial, sometimes cannot be pursued due to economic considerations arising out of the time and effort involved.

Requisite in the development of new coating materials is the use of a particular coating method as well as consideration of holdover or carryover effects. Holdover effects result in the contamination of one candidate coating material due to residual coating material remaining in the coat dispensing apparatus and/or coat-receiving substrate from a prior test coating material. Contamination as a result of holdover effects are generally additive and provide a level of error in coat formulation that is difficult to control. It is therefore preferable, especially when the volume of coating material to be tested is small, to use a coating method that either eliminates or significantly reduces holdover effects. Use of a disposable method for dispensing as well as receiving the test coat material would eliminate problems associated with holdover effects.

A variety of methods for coating desired substrates or materials are available and include spin coating, die coating and non-contact jet coating methods. Spin coating is a technique commonly used in the field of electronics where the coat material is dispensed onto a desired surface by centrifugal force (spinning). The coatweights resulting from this method are limited to very thin coatings and there is a significant loss of material during the coating process. In both the die coating and non-contact jet coating methods, die and jetting nozzle costs prohibit their modification to disposable units. Prior to the instant application, an inexpensive, efficient and disposable method for testing a large number of coating materials has not been known. While many significant advances in coating technology have been made in recent years, acceleration of the rate at which coating materials can be identified, screened, investigated and optimized will be recognized as a desirable goal by those skilled in the art.

Summary of the Invention

An object of the invention is to provide a multi-well apparatus for making arrays of coating materials. Such arrays are suitable for analysis and may comprise a disposable two-layer assembly where the first layer contains a plurality of wells and the second layer is a substrate layer. Both layers can be flexible, with the second or bottom layer being detachable from the overlying first layer. Such an apparatus can be made of disposable material, thus providing a cost-effective, efficient and reliable means of making and testing numerous formulations of coating material.

The invention also provides a method of developing a new coating having a desired performance characteristic with regard to a property of a coating, comprising: a) providing an array of coating wells, b) placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coatings having different parameter values in a plurality of coating wells; c) correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array; d) applying a leveling force to the array of wells to level the coating material in the coating wells; and e) testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material, whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating. Optionally, the coatings in the array can be dried while the leveling force is applied. The above combinatorial, high-throughput method of screening candidate coat materials results in a significant increase in the discovery rate of new coating materials. In a preferred embodiment the leveling force may be provided by a centrifuge.

Further features, details, and advantages of the invention will be more apparent with reference to the following detailed description, taken in conjunction with the accompanying drawings, which illustrate, by way of example, principles of the invention.

Brief Description of the Drawings

FIG. 1 is a generic schematic of the combinatorial discovery process;

FIG. 2 is a perspective view of an example of a robotic dispenser usable in one embodiment of the invention;

FIG. 3 is a top view of an example of a well plate usable in one embodiment of the invention;

FIG. 4 is a perspective view of an example of a well plate having a removable well bottom, comprising a substrate to which sample coatings are applied, usable in one embodiment of the invention;

FIG. 5 is a perspective view of another example of a well plate having a removable well bottom comprising a substrate to which sample coatings are applied, usable in one embodiment of the invention;

FIG. 6 is a side view of a well plate having a curved bottom usable in one embodiment of the invention;

FIG. 7 is a side view of a flexible well plate having a removable top portion usable in one embodiment of the invention;

FIG. 8 is a side view of a well plate having a laminate construction usable in one embodiment of the invention;

FIG. 9 is a schematic diagram showing leveling of coating array materials by application of a leveling force and curing by hot air;

FIG. 10 is a perspective view of an example of a centrifuge usable in an embodiment of the invention;

FIG. 11 is a perspective view of an example of a swing arm centrifuge rotor assembly usable in one embodiment of the invention, showing the assembly loaded with well plates; and

FIG. 12 is a perspective view of an example of a 96-well plate usable in one embodiment of the invention.

Detailed Description of the Preferred Embodiments

In accordance with one aspect of the invention, it has been recognized that by using automation of certain development processes, miniaturization of samples to be tested, database development and manipulation, and using algorithms to identify candidate materials from information contained in databases, one can increase the number of coating materials that can be developed to meet identified needs. As used herein, the term "combinatorial" refers to the combined approach of high-throughput analysis of libraries consisting of arrays of coat material formulations. Included in the high-throughput analysis are automated or robotic processing of the sample arrays.

Combinatorial methods have been used in the medical , pharmaceutical and biotechnology industries to develop chemical compositions, particularly pharmaceuticals and medicaments, for a number of years. However, these prior combinatorial methods have not been well suited to development of new coatings. Applicants herein provide techniques for generating arrays of coating formulations, well suited to the application of combinatorial chemistry methods. These techniques allow new coatings to be screened and evaluated on a high throughput basis, in order to produce new coatings economically.

Combinatorial Approach

With reference to FIG. 1 of the drawings, which are given by way of example, and not by way of limitation, a system 10 in accordance with principles of the invention comprises a method of developing new coatings by means of a combinatorial approach. A first step 12 is to define

what end result coating is desired, and what characteristics and qualities such a coating will have. To achieve the desired result a new material, or a new construction of several materials, such as a laminate for example, comprising new and/or conventional materials combined in a novel way may be required.

At the outset it should be understood that combinatorial methods can be applied to both the process of creating coating materials by formulation or synthesis, and to creating coating parameters or desired characteristics.

Returning to consideration of one example of a combinatorial approach to coating development, the next step 13 is to select likely candidate materials. These can comprise formulations of generally dilute solutions of raw material ingredients 14 that are contemplated as likely elements or components that may provide a coating material with desired characteristics. In the next step 16 a material library of a few to a few hundred thousand, or more, chemical combinations are formed and dispensed into an array of coating wells 18 using a robot or other automated device 17 to make a library or array of coating materials . Incidentally, the "libraries" may include the samples in a single array, or the samples may form a plurality of arrays, processed either concurrently or successively. The chemical combinations forming at least part of the library are then processed in parallel as indicated at reference numeral 19. Processing can include exposing the coating array to a variety of processing variables such as heat, and time as well as applied leveling forces to shape the resultant library or array of coat samples, as can be accomplished, for example, by a centrifuge 20. In the next step 21 high throughput analysis is performed whereby the library is screened by detectors that quickly scan various properties of the coating materials. After the high throughput analysis, materials with the desired properties are identified 22 with the results entered into a large database 23, allowing up to 25,000 variations of materials to be tested at one time. Each library is comprised of one or more arrays of variations of materials to be tested. Each individual site in an array will correspond to a specific formulation of a coat material, wherein the parameter or coat descriptor(s) of the material located at that site is known. Miniaturization of the sample size facilitates processing and greatly saves cost and time thereby increasing efficiency and the rate of discovery. The end result is discovery and determination of the most successful new material(s) and the process or parameters used to produce the new materials. These materials are then selected for large scale production and commercialization 24.

The combinatorial approach to development and testing of novel coat materials greatly benefits from use of devices and apparatus that allow flat coating samples in the arrays or within

wells in the arrays. Additional embodiments encompassing such devices and apparatus are included in the present invention and further described below.

When trying to coat one formulation after another in a rapid fashion, "holdover" considerations are important. As used herein, the term "holdover" is defined as the volume of material that is residual in a cavity after it is emptied and could contaminate the next batch of material deposited into the cavity. As volumes of the cavity get smaller, the potential for holdover increases. For example, tubes, pipette tips, material dispensers and such all have potential holdover volumes. The contamination is also a function of the rheological nature or viscosity of the material that is deposited into the cavity. Holdover effects in traditional methods of developing coating materials greatly increases the level of error, compromising the identification of correct parameters of a new coat material. In the present invention, holdover and its contaminating effects are eliminated by use of a disposable dispensing device 25 (FIG. 2) and a disposable substrate assembly (formatted as a multi-well apparatus) 26, both of which are further described below. As used herein, the term "substrate" is defined as any coat-receiving surface or material, or a substance upon which a sample coat material resides which allows the testing of that sample. A "substrate assembly" is a composite of materials formed into a unit or apparatus for holding a large number of different coating samples in an array format (FIG. 3). An "array format" as used herein, is a matrix format where the samples of coating material are arranged as discrete coated areas 31 on a surface, such as a planar surface. For example, a 48-well coating array (FIG. 3) would have 48 discrete coated areas arranged as 6 rows 27 and 8 columns 28.

Multi-well Apparatus for Parallel Processing of a Material Library

An initial step in the development of a coating is to create the various mixed formulations to be placed in the wells in the array. In one embodiment of the present invention, such sample formulations can be mixed or prepared in a multi-well plate format (FIG. 3) with each individual well containing a unique, pre-defined formulation to be tested. A variety of types of commercially available multi-well plates suitable for use in the present invention can be used (Millipore Corp., Polyfiltronics, VWR Scientific). Such multi-well plates can vary in size of plate dimension, size of well (outer circumference as well as well-depth), type of material used to construct the multi-well plate (for example, polystyrene or polypropylene, rigid plastic or flexible plastic). The biotechnology and pharmaceutical industry utilizes multi-well plates (generally 48-, 96- or 256-well plates) whose outer dimensions are standardized for use with robotic dispensers. Generally, standardized multi-well plates are rectangular, rigid, stackable

plates with right edges of the top or lid portion being curved 29. The outside dimensions of a complete multi-well unit are approximately 5 x 3.25 inches. Such multi-well plates are suitable for use in the present invention. In general, the well size used should be of substantial volume so as to allow adequate robotic mixing of the required or needed amount of each formulation without drying up of the solutions contained in the wells. Preferably a well volume of .5 to 3 cubic centimeters in volume is contemplated for use in the present invention. The minimum quantity or volume of sample to be mixed in a "mother" wellplate will vary depending upon the desired coating thickness, domain size and formulation of the coating solution.

As used herein, a "mother" well plate is defined as a source well plate. For example, a 25 micron thick coating that is 1 cm^2 in domain size with a coating solution that is 50% solids, will require (1 cm^2 x 25 microns / 0.5) volume units or 0.0050 cc of solution. "Domain size" as used herein, refers to the minimum area required for the coated sample as determined by downstream testing. The appropriate volume of individual formulations from this mother well plate can then be dispensed to a sample or "daughter" well plate to make a coating with the desired domain size for subsequent analysis and data collection. It should be understood, that alternative embodiments include use of a single well plate as both the mother and daughter well plate. In such a case, the well plate into which the sample formulations are mixed will also serve as the well plate from which the coating materials will be tested. Again, considerations of desired coating thickness, domain size and formulation of coating solutions will be included in determination of minimum volume of well size required. Additional embodiments of well plate apparatus design will be discussed further below.

Automated Dispensing of Candidate Coat Materials for Testing

A disposable metering device can be used to dispense the formulations from a mother well plate to a daughter well plate. A robotic dispenser (available commercially for example, from Hamilton Zinser Packard) (FIG. 2) is one such device. Robotic dispensers allow for rapid and automated dispensing of a specified quantity of a large number of samples. The well plate format to be used for the daughter well plate will also depend on the domain size requirement of the coating. For example, a 6-, 12-, 24-, 48-, 96-, or 384- well plate format are commercially available formats which can be used in the present invention with the commercially available robotic dispensers. The robotic dispenser will have a platform area upon which the substrate well plates reside (FIG. 2; "A").

Alternatively, in the case where a single well plate is used as both the mother and daughter wellplate, a robotic device can also be used for mixing as well as dispensing component

materials for the sample coating formulation to be tested. Such a device could have multiple dispensing units 30 from which specific and precise amount of an individual component is dispensed into a single well. The sample solution can be dispensed using disposable pipette tips 30b attached to the pipettors 30c. For example, a separate dispensing unit for each component can be used to dispense the appropriate amount of a respective component into a single sample well. Such a dispensing unit can be disposable which will allow rapid and accurate automation of the combinatorial method for formulating or synthesizing a new coating with elimination of holdup or contamination problems. Examples of disposable dispensing units include, polyethylene or other type of tubing and disposable pipette tips.

Alternative Designs of Multi-well Apparatus for Parallel Processing

Alternative embodiments of well plate design include providing a two-piece coating well apparatus having at least a substrate portion 32 and a multi-well or sample-containing template 34 which can be separated from one another (FIG. 4). Once leveled and dried, the coating material 36 is held by the substrate portion 32 of the assembly. This type of well plate assembly is designed such that the base substrate-portion (or bottom half of the assembly) 32 can be removed from the multi-well template portion 34 of the well plate assembly. Various embodiments of a well plate design having a removable bottom are contemplated and further described below. FIG. 5 shows an example of a multi-well plate depicting the array format useful in the invention. Coating material samples are placed within the apertured, multi-well template top 47. Such multi-well plates will form an array 41 or library format of the different formulations as discrete coated areas 40 on a planar substrate sheet 42. A multi-well plate with a removable top or cover can also be used as a well plate assembly. An example of such a multi-well plate design is shown in FIG. 7. The well plate design can also include modifications to the well plate to prevent distribution of coating material onto the inner walls of the wells. For example, a release coating can be applied to the inner walls 43 of the wells to prevent any sample material from moving up and onto the well walls during application of a leveling force.

An additional embodiment of the present invention includes multi-well plates designed to obtain flat coatings in all of the wells of assembly. Current commercially available multi-well plates have a flat-bottom surface for the entire plate. This results in an uneven distribution of sample material in the wells located along the perimeter of the multi-well plate 68 when current swing arm type of centrifuge rotors 70 are used to apply a leveling force. FIG. 6 shows an example of a modified multi-well plate designed to obtain flat coatings in all of the wells. Such a well plate will have a curved base plate 44 where the curvature of this base is parallel to the

circumference of the centrifuge rotor, or is curved so as to substantially match the curvature of the curvilinear path of the well plate during centrifugation. With a curved-bottom well plate 44, sample material or coating solutions in all of the wells, including perimeter wells 45, will be at the same distance from the spin axis of the centrifuge. Thus, coating material in all of the wells will have a flat distribution following centrifugation. The top view of such a multi-well plate can be as depicted in FIG. 5. A flexible substrate and apertured well plate may be employed to provide a curved configuration when mounted in a centrifuge.

A specialized laminate well plate construction is also envisioned as an alternative embodiments of the present invention. FIG. 8 shows a cross sectional view of a representative laminate multi-well plate assembly. In one case, the assembly is made up of at least 4 layers and is shown in FIG.8. The top or first layer 46 corresponds to the multi-well or sample holding portion of the assembly. This layer need only be thick enough to provide a sufficient barrier between adjacent wells so that the dispensed coating material 50 does not cross contaminate adjacent samples. Where a very small amount of coating material 50 is to be tested, this layer need not be very thick and could be made of, for example, thin plastic, foam or paper with each well formed of holes placed in linear, multiple rows to form an array pattern. Preferably, the top layer will be about .01 to about 1 mm, or about1 to about 10 mm, or about 1 to about 5 cm in height. This top layer 46 can be coated with a Pressure Sensitive Adhesive (PSA) (not shown) to attach it to the substrate layer 48. This will also help to seal the wells so that cross-contamination of sample coating material from one well does not mix with its neighbors. The second layer is the substrate layer 48 and can be formed of a variety of materials, such as plastic, polymeric resin or paper, so long as it will hold the sample coating material 50 in a flattened manner. The second layer will preferably be about 1 to about 100 microns, or about 1 to about 10 mm, or about 1 to about 5 cm in thickness. The third layer is a Pressure Sensitive Adhesive layer (PSA) 52. The PSA layer 52 can be about 5 to about 30 μm , or about .005 to about .03 mm, or about.0005 to about.003 cm in thickness depending upon the type of adhesive and degree of adhesion desired. The fourth layer is a liner 54 coated with a release layer such as silicone, which can be removed or peeled away from the PSA layer 52 leaving the adhesive on the bottom of the substrate layer as the new bottom layer. This type of multi-well plate design is suitable for example, where the stickiness or tackiness of a coating material is to be tested. In such a case, it is desirable to have an array library which will remain stationary or adhere to a support surface by the PSA layer 52 while each individual coating sample is tested. Use of the PSA 52 on the layer 48 will allow the array library to remain stationary and not lift up during testing.

Leveling Force

Once the different formulations are dispensed into a multi-well plate assembly 63, the coat formulations are made into flat coatings 64 within the wells by use of a leveling force. A "leveling force" as used herein, is defined as any force sufficient to cause a sample or coat material to distribute evenly and flatly onto a substrate. A leveling force will also remove any residual air bubbles present within the sample coat formulation. A variety of leveling forces are contemplated for use in the present invention including, for example, use of centrifugal force, use of a vacuum or negative pressure force, use of an electrostatic force, or use of a magnetic force. In the case where magnetic leveling force is used, the test coat formulation will contain magnetic particles, powder, or a compound such as ferrite, that is responsive to a magnetic force. Use of a leveling force need not be limited to single-coat assessments. Where the processing of a multi-layer construction of coat material is desired, a leveling force can be repeatedly applied following dispensing of individual layers of a coat to be tested. The final array obtained will be a planar sheet containing discrete areas in a grid format of multi-layer coat formulations.

FIG. 11 shows an example of a centrifuge that can be used for applying a leveling force to a multi-well plate. Such swing arm-type centrifuges with multi-well plate holders (FIG. 12) are available commercially (for example, VWR Scientific, "MicroPlus GH 3.8 rotor centrifuge). The rotor for use in such a centrifuge is designed so as to hold an even number of multi-well plate assemblies. The multi-well plate assemblies 68 are loaded into the rotor 70 in an upright or horizontal position. During centrifugation, the plates are directed into a vertical position which then levels or flattens the sample formulations onto the substrate layer. After the formulations are dispensed in a multi-well plate assembly, the assembly is placed in a swing-arm centrifuge and the coatings are spun at controlled speeds so as to form a flat coating within each well 64. For example, with a standard centrifuge, a 10-min. spin at 2000 rpm will be sufficient to evenly distribute the coat materials within each well. There is no loss of sample material with use of a swing-arm centrifuge.

Additional methods of casting sample coat formulations include those which can also simultaneously dry the coating material during casting. For example, a centrifuge which has been modified to hold circulating hot air or other gas which will aid in the evaporation of carrier solvents in the coating formulations is also contemplated for use in the present invention and is diagrammed schematically in FIG. 9. The hot air 66 circulating over the formulations during centrifugation aids in the drying of the coating by evaporation of volatiles or solvents. As with a centrifuge, devices used to provide alternative methods of applying a leveling force can also be modified so as to simultaneously dry the coat formulations. For example, an apparatus utilizing

a vacuum or electrostatic force as the leveling force can be modified to circulate hot air and include alternate arrangements for drying.

High Throughput Analysis, Data Storage, Data Modeling and New Materials Discovery

The above methods provide an array 40 of coating materials with each site in the grid array containing a coat material having a known parameter which differs from parameter values of the materials contained on the other sites (FIG 1; step 16). With this array, the plurality of coating materials can each be tested for performance of each coating. Because the parameter value of the coating contained at each site is known, the value of a parameter associated with a desired performance of a coating can be determined. All information obtained by this high throughput analysis screening a coat material library are then entered into a database. From this database identification of the most successful new coat materials and the parameters and descriptors used to produce them is achieved (FIG. 1, step 23). Such a database will also serve as a storage library to aid in the formulation of future parameters to characterize the coatings.

Example I

This example demonstrates the use of a multi-well plate combined with a centrifugal leveling force for estimation of coat weight of a sample coat material formulation. This example is intended to be representative of one embodiment of the invention, and not intended as limiting the scope of the invention.

The emulsion polymer formulation used was S-2000. S-2000 is a nondispersable emulsion acrylic polymer manufactured by Avery Dennison Corporation, Pasadena CA in accordance with U.S. Patent No. 5,221,706. A 96-well plate obtained from Polytronics was used as a daughter well plate. The well plate remained flat during centrifugation. Each well contained an equivalent sample material formulation for determination of coat weight.

Diameter of each well = 0.6 cm

Cross-section of each well = 3.14 x 0.6 cm2 = 1.884 cm2

Weight of coat material in E7 position of array = 0.0153 gm

Wet coat weight in E7 = 0.0153/.0001884 = 81.21 gsm

% solids in wet solution = 52.1%

Dry coat weight in E7 = 42.3 gsm

Results:

The emulsion did not dry fast and remained opaque. Hence the need for higher temperature drying. Material in wells located on the perimeter wells did not level evenly. Coat material dispensed into the center wells were centered and evenly flattened in the horizontal direction. The uneven leveling observed in the perimeter wells is believed to be a result of the centrifugal force acting at an angle to the bottom of the well, unlike the preferred flexible configuration of Fig. 6.

This example demonstrates the utility of using a multi-well plate combined with a leveling force for high-throughput analysis of specific parameters or characteristics of coat material formulations in an individualized manner.

The invention has been described with reference to various specific and preferred embodiments and techniques. However, it should be understood that many variations and modifications may be made while remaining within the spirit and scope of the invention

CLAIMS

1. A method for efficiently preparing a large number of sample coatings comprising the steps of:

(a) forming a series of sample receptacles or wells by providing a flexible substrate and an overlying apertured sheet with the apertured sheet in tight sealing engagement with the substrate;

(b) applying different samples of material in liquid form into said receptacles;

(c) placing said flexible substrates with said sample receptacles thereon in a centrifuge;

(d) activating said centrifuge with said receptacle mounted therein to flatten out the sample material in said receptacles, with the centrifugal force acting perpendicular to the bottom of the receptacles;

(e) drying said samples while they are within the centrifuge; and

(f) removing the apertured plate to leave the samples exposed on said substrate.

2. A method as defined in claim 1 wherein said applying step involves the application of various adhesive compositions into said receptacles or wells.

3. A method as defined in claim 1 wherein multilayer samples are formed by repeating steps (b) through (e) prior to step (f).

4. A method as defined in claim 1 wherein an array of at least four wells are formed.

5. A method as defined in claim 1 wherein hot air is applied to the samples during centrifugation.

6. A method as defined in claim 1 wherein said substrate is formed of paper.

7. A method of forming a test coating comprising the steps of:

forming a receptacle for receiving a material sample, said receptacle having a flat bottom and enclosing sides:

depositing a fluid material sample in said receptacle;

mounting said receptacle in a centrifuge with the outward centrifugal force being perpendicular to the bottom of said receptacle;

activating said centrifuge to flatten the material in the receptacle; and

drying said material while the sample is being rotated and flattened by the centrifugal action.

8. A method for efficiently preparing a large number of sample castings comprising the steps of:

forming a series of sample receptacles by providing a substrate and an overlying apertured sheet with the apertured sheet in tight sealing engagement with the substrate;

applying different samples of material in liquid form into said receptacles;

drying said samples; and

removing said apertured sheet to leave said material samples on said substrate.

9. A method as defined in claim 8 including the step of applying force to said samples perpendicular to the bottom of said receptacles to flatten out said samples

10. A multi-well apparatus for providing an array of coating material suitable for analysis of performance characteristics comprising:

an at least 2-layer assembly comprised of a top and a bottom layer, wherein the top layer comprises an apertured sheet;

the bottom layer comprises a substrate layer for receiving the coating material; and

the bottom layer is detachable.

11. A multi-well apparatus as defined in claim 10 wherein the multi-well apparatus is flexible.

12. A method of testing coating materials, comprising the steps of:

providing an array of coating wells, each well being configured for receiving a coating material having a known composition;

placing a coating material having a known composition in each coating well, varying the composition so as to provide a plurality of coating materials having different compositions in a plurality of coating wells;

correlating the composition of the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a specific composition is associated with each coating well position in the array;

placing said coating wells with said compositions into a centrifuge, and activating said centrifuge;

drying said coating materials; and

testing the resultant coatings.

13. The method of claim 12 including providing wells in the form of a flexible substitute and a flexible overlying apertured sheet.

14. The method of claim 12 including the step of heating said coating materials while said centrifuge is activated.

15. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter; said array of coating wells comprising a substrate and an overlying apertured sheet;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

drying said coating samples; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

16. The method of claim 15, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after drying, whereby the coating material array is carried by the substrate alone after separation.

17. The method of claim 15, wherein the well depth and volume is substantially greater than that of the coating volume.

18. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

applying a centrifugal force to the array of coating wells to level the coating material in the coating wells;

curing said coating samples under said coating leveling force; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

19. The method of claim 18, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after application of the leveling force, whereby the coating material array is carried by the substrate alone after separation.

20. The method of claim 12, further comprising the steps of:

curving the said array of coating wells to substantially match the curvature of the curvilinear path of the array during centrifuging.

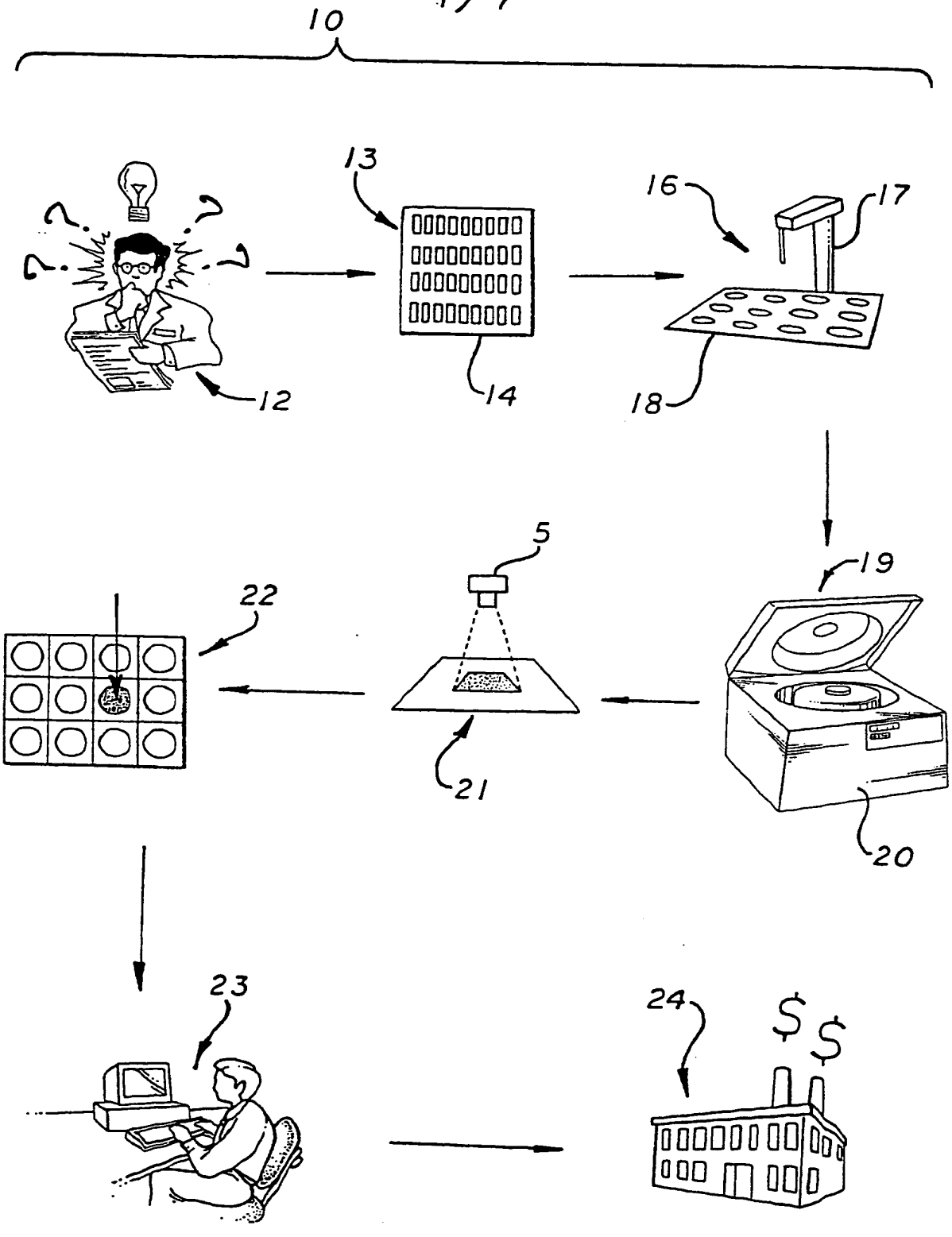


FIG. 1

FIG. 2

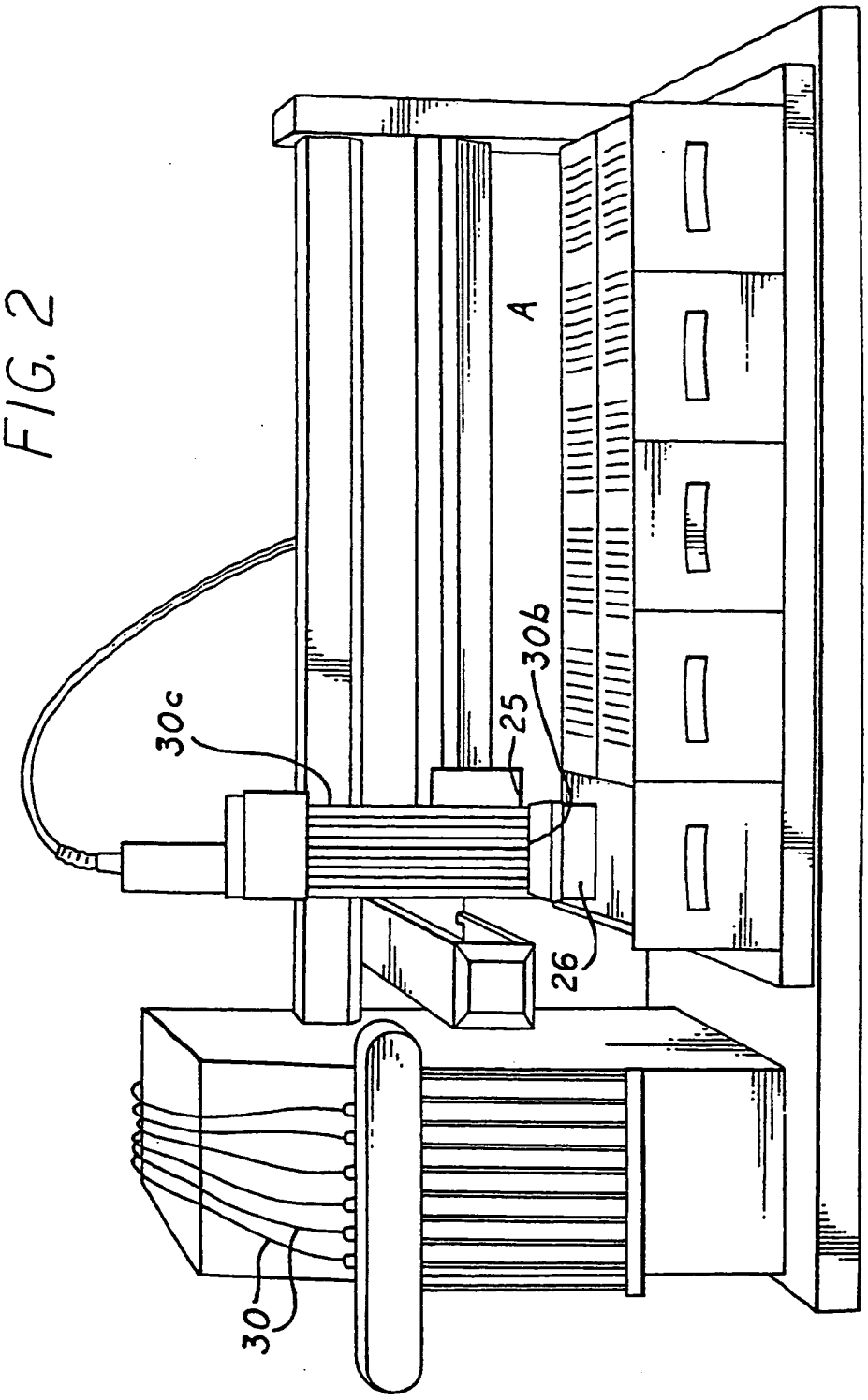


FIG. 3

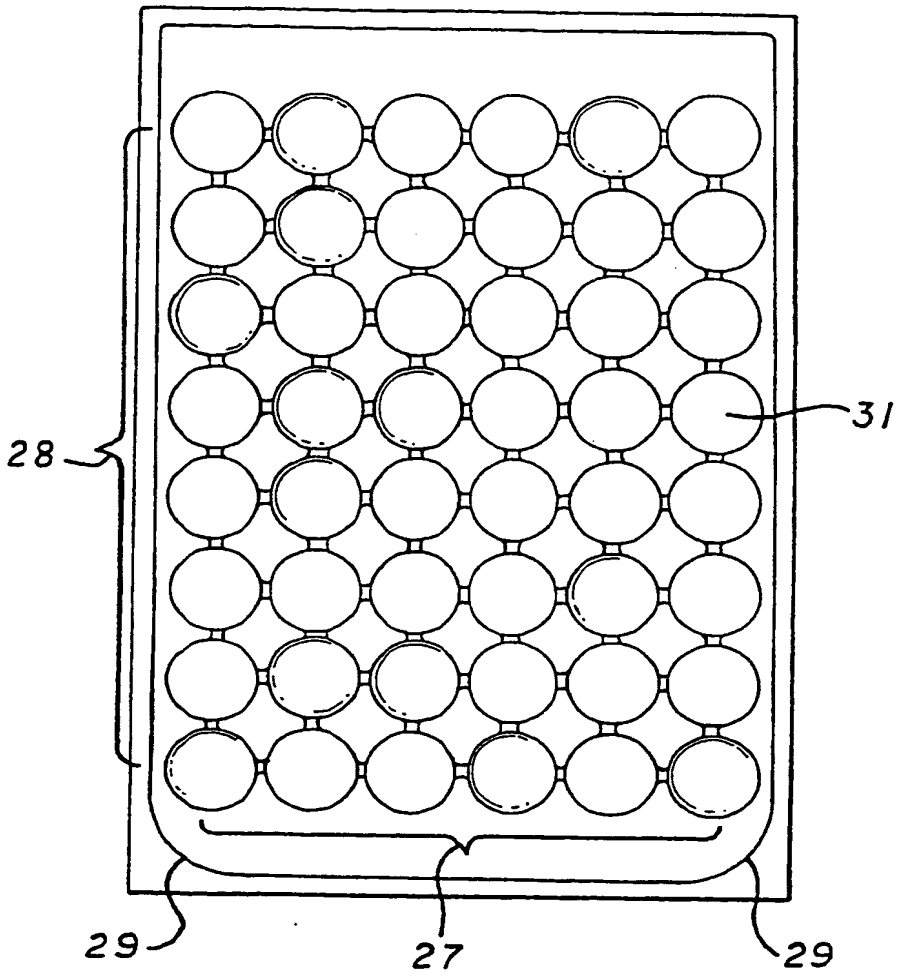


FIG. 4

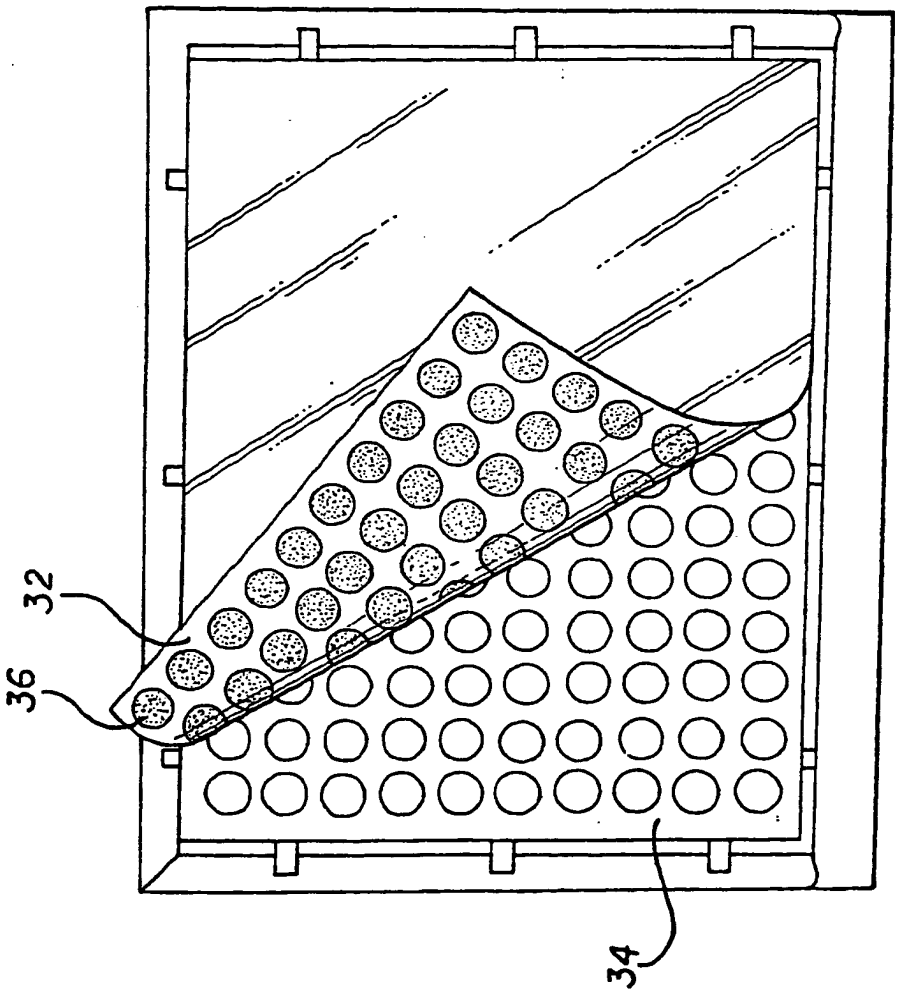
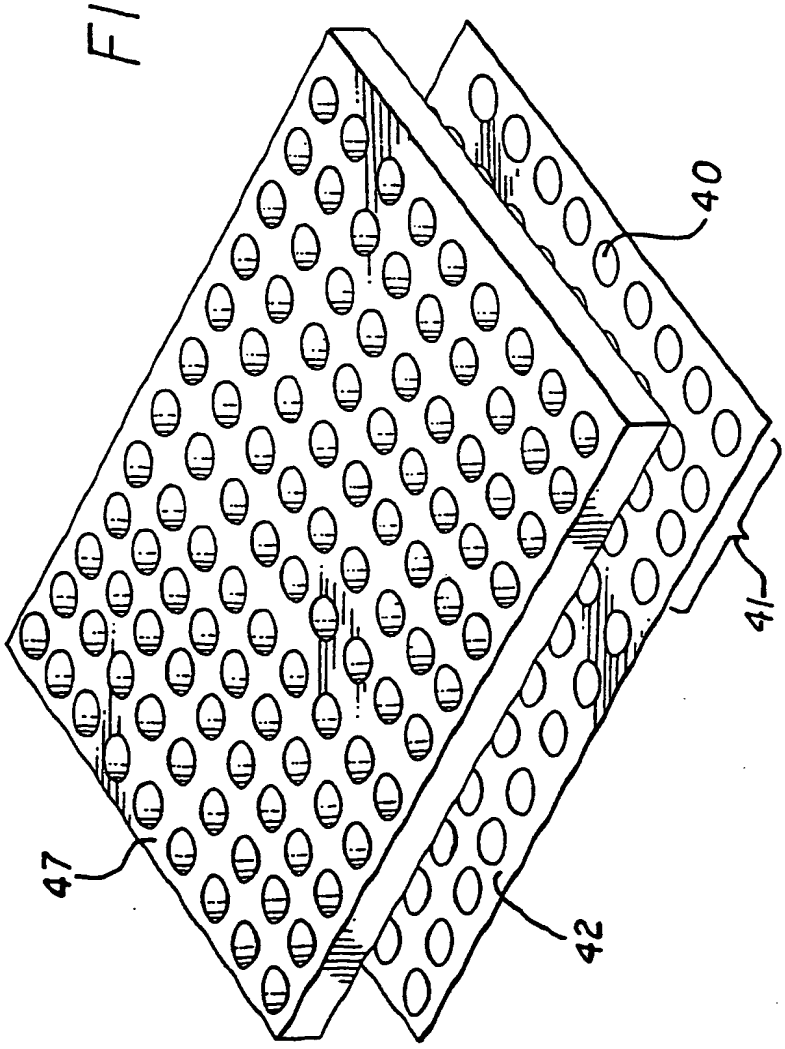


FIG. 5



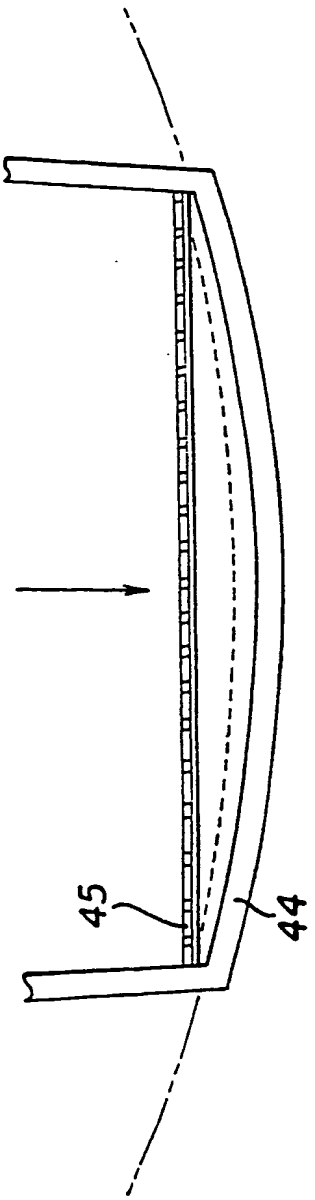


FIG. 6

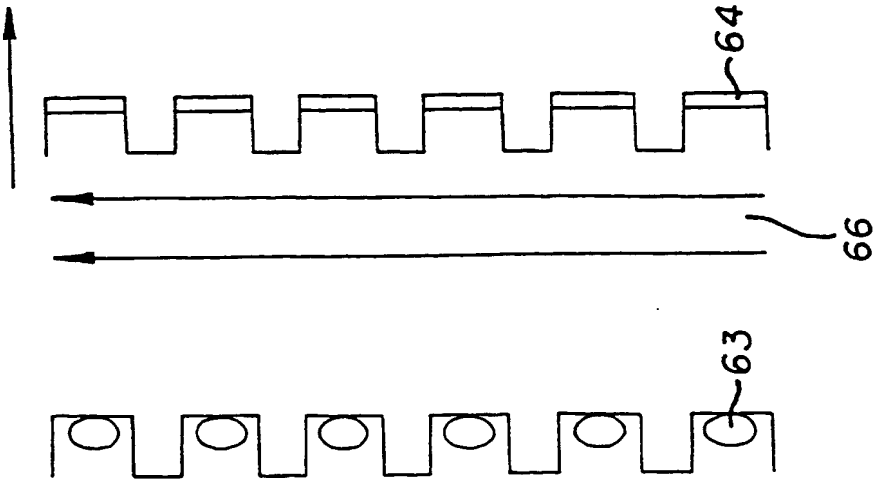


FIG. 9

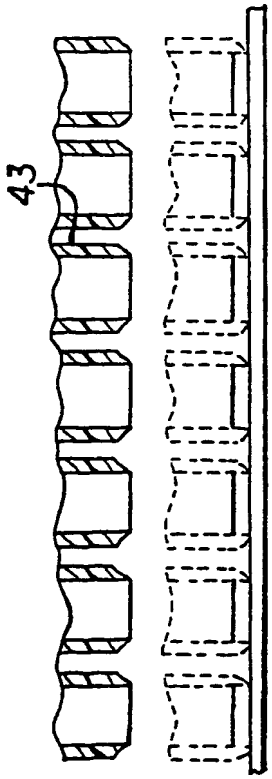


FIG. 7

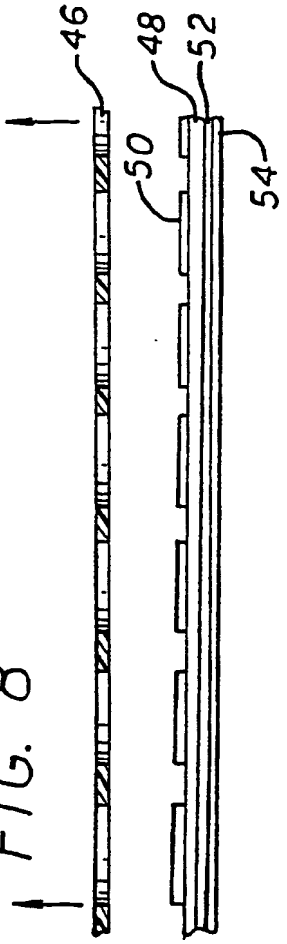


FIG. 8

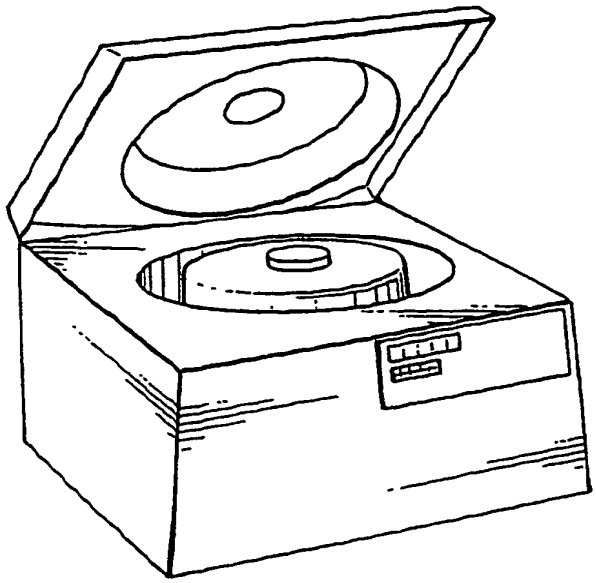
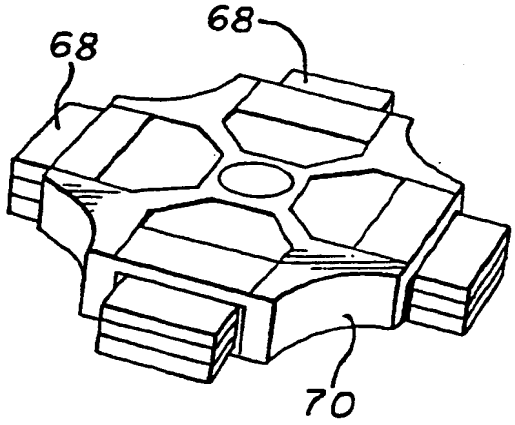


FIG. 10

FIG. 11



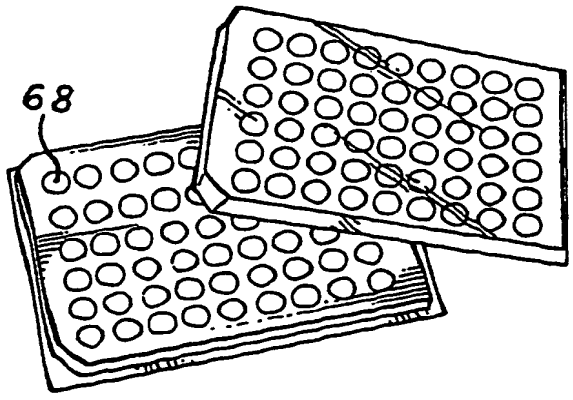


FIG. 12

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

A. CLASSIFICATION OF SUBJECT MATTER
IPC(7) :G01N 31/00, 1/28; C12M 1/34
US CL :422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X - Y	US 4,299,920 A (PETERS) 10 November 1981, see entire document.	10-11 --- 1-9,12-20
Y	Chemical Abstracts, Vol. 94, No. 16, issued 20 April 1981, R. A. Machevskaya et al, "Study of the interrelation of Propeties of Coatings and the Composition of Epoxy-Phenol Compositions" see page 93, Col. 1, abstract No. 123179p, Lakokras. Mater. Ikh Primen., 1981, (1), 35-36.	1-20
Y,E	US 5,985,356 A (SCHULTZ et al) 16 November 1999, see entire document.	1-20
A	CA 2,260,807 A (EIPEL et al) 29 January 1998.	1-20

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
18 JANUARY 2001	31 JAN 2001
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer ARLEN SODERQUIST Jean Proctor Paralegal Specialist Telephone No. (703) 308-0661

Form PCT/ISA/210 (second sheet) (July 1998)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 363,504 (PPG HELLIGE B.V.) 18 April 1990.	1-20

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

B. FIELDS SEARCHED
Electronic data bases consulted (Name of data base and where practicable terms used):

STN search in CA file
search terms: coating or adhesive or protect?, layer, film, combinator?, library, centrifug?, dry dried drying, cure, curing, cured

PCT POWER OF ATTORNEY

Printed on 07.12.2000 11:57:19 AM

0-1	PCT Power of Attorney (for an international application filed under the Patent Cooperation Treaty) (PCT Rule 90.4)	
0-1-1	Prepared using	PCT-EASY Version 2.91 (updated 01.07.2000)
1	The undersigned applicant(s)	AVERY DENNISON CORPORATION
1-1-1	hereby appoints (appoint) the following person	Att: Alan C. Rose OPPENHEIMER WOLFF & DONNELLY LLP 2029 Century Park East, Suite 3800 Los Angeles, CA 90067-3024 United States of America
1-2	as	agent
1-3	to represent the undersigned before	all the competent International Authorities
1-4	in connection with the international application identified below:	
1-4-1	Title of the invention	AN APPARATUS FOR HIGH THROUGHPUT PRODUCTION OF COAT MATERIAL ARRAYS, AND ANALYTICAL METHODS USING
1-4-2	Applicant's or agent's file reference	310048-488WO
1-4-3	International application number (if already available)	PCT/US00/29854
1-4-4	filed with the following Office as receiving Office	U.S. Patent and Trademark Office
1-5	and to make or receive payments on behalf of the undersigned.	
2-1	Signature of applicant	AVERY DENNISON CORPORATION
2-1-1	Name	Arthur B. Moore, Esq., Chief Patent Counsel
3	Date	

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

A. CLASSIFICATION OF SUBJECT MATTER
IPC(7) :G01N 31/00, 1/28; C12M 1/34
US CL :422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 422/102, 104; 435/288.2, 288.3, 288.4; 436/2, 174, 183

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X - Y	US 4,299,920 A (PETERS) 10 November 1981, see entire document.	10-11 --- 1-9,12-20
Y	Chemical Abstracts, Vol. 94, No. 16, issued 20 April 1981, R. A. Machevskaya et al, "Study of the interrelation of Propeties of Coatings and the Composition of Epoxy-Phenol Compositions" see page 93, Col. 1, abstract No. 123179p, Lakokras. Mater. Ikh Primen., 1981, (1), 35-36.	1-20
Y,E	US 5,985,356 A (SCHULTZ et al) 16 November 1999, see entire document.	1-20
A	CA 2,260,807 A (EIPEL et al) 29 January 1998.	1-20

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search
18 JANUARY 2001

Date of mailing of the international search report
31 JAN 2001

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Form PCT/ISA/210 (second sheet) (July 1998)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/29854

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 363,504 (PPG HELLIGE B.V.) 18 April 1990.	1-20

INTERNATIONAL APPLICATION

WITH ANNEXES TO THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT INCORPORATED

An Apparatus for High-Throughput Production of Coat Material Arrays, and Analytical Methods Using such Arrays

Related Patent Application

This application claims priority from prior U. S. Provisional Patent Application Serial Number 60/162,349 filed October 29, 1999, the disclosure of which is hereby incorporated by reference.

Background:

1. Field of the invention:

The invention relates generally to methods and apparatus for identification and optimization of coating materials and properties for desired applications. More specifically, the invention relates to an improved process of creating coatings, involving identifying candidate materials and screening and optimizing formulations and coating parameters for desired applications.

2. Description of related art

Development of coating materials, for example adhesive coatings, release coats, protective coatings, and the like as well as films and laminate constructions of layered materials, has conventionally been a time consuming and labor intensive process. Candidate materials are identified primarily based on knowledge and experience with what compositions have worked before in related applications and investigating like materials and combinations of materials. This usually involves preparing a coating formulation, preparing a test coating for evaluation (often involving several tries to attain the desired parameters such as coat weight, cure, etc. for evaluation), drying the coating, then evaluating the coating by testing the property of interest, such as permeability, tack, shear and bending strength, surface roughness, etc., and entering the results in a database for comparison with further coatings to be developed and tested. Problems of cross-contamination and holdover further limit the number of formulations that can be screened in a given time period. This is a time-consuming process and as a result one skilled in the art, even with support staff to assist and carry on tasks in parallel, has conventionally been able to screen at most a few coatings per day, most often only one or two.

Because of the lengthy time required to screen and then investigate candidate materials and associated coating application parameter values to select and optimize coatings, those skilled in the art generally must focus on families of materials known to possess properties likely to prove successful in the intended use. Investigation of unconventional or simply previously

untried materials is usually limited. Moreover, development of coating materials for a particular application is also a time-consuming process, and development of new coatings, while potentially beneficial, sometimes cannot be pursued due to economic considerations arising out of the time and effort involved.

Requisite in the development of new coating materials is the use of a particular coating method as well as consideration of holdover or carryover effects. Holdover effects result in the contamination of one candidate coating material due to residual coating material remaining in the coat dispensing apparatus and/or coat-receiving substrate from a prior test coating material. Contamination as a result of holdover effects are generally additive and provide a level of error in coat formulation that is difficult to control. It is therefore preferable, especially when the volume of coating material to be tested is small, to use a coating method that either eliminates or significantly reduces holdover effects. Use of a disposable method for dispensing as well as receiving the test coat material would eliminate problems associated with holdover effects.

A variety of methods for coating desired substrates or materials are available and include spin coating, die coating and non-contact jet coating methods. Spin coating is a technique commonly used in the field of electronics where the coat material is dispensed onto a desired surface by centrifugal force (spinning). The coatweights resulting from this method are limited to very thin coatings and there is a significant loss of material during the coating process. In both the die coating and non-contact jet coating methods, die and jetting nozzle costs prohibit their modification to disposable units. Prior to the instant application, an inexpensive, efficient and disposable method for testing a large number of coating materials has not been known. While many significant advances in coating technology have been made in recent years, acceleration of the rate at which coating materials can be identified, screened, investigated and optimized will be recognized as a desirable goal by those skilled in the art.

Summary of the Invention

An object of the invention is to provide a multi-well apparatus for making arrays of coating materials. Such arrays are suitable for analysis and may comprise a disposable two-layer assembly where the first layer contains a plurality of wells and the second layer is a substrate layer. Both layers can be flexible, with the second or bottom layer being detachable from the overlying first layer. Such an apparatus can be made of disposable material, thus providing a cost-effective, efficient and reliable means of making and testing numerous formulations of coating material.

The invention also provides a method of developing a new coating having a desired performance characteristic with regard to a property of a coating, comprising: a) providing an array of coating wells, b) placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coatings having different parameter values in a plurality of coating wells; c) correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array; d) applying a leveling force to the array of wells to level the coating material in the coating wells; and e) testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material, whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating. Optionally, the coatings in the array can be dried while the leveling force is applied. The above combinatorial, high-throughput method of screening candidate coat materials results in a significant increase in the discovery rate of new coating materials. In a preferred embodiment the leveling force may be provided by a centrifuge.

Further features, details, and advantages of the invention will be more apparent with reference to the following detailed description, taken in conjunction with the accompanying drawings, which illustrate, by way of example, principles of the invention.

Brief Description of the Drawings

FIG. 1 is a generic schematic of the combinatorial discovery process;

FIG. 2 is a perspective view of an example of a robotic dispenser usable in one embodiment of the invention;

FIG. 3 is a top view of an example of a well plate usable in one embodiment of the invention;

FIG. 4 is a perspective view of an example of a well plate having a removable well bottom, comprising a substrate to which sample coatings are applied, usable in one embodiment of the invention;

FIG. 5 is a perspective view of another example of a well plate having a removable well bottom comprising a substrate to which sample coatings are applied, usable in one embodiment of the invention;

FIG. 6 is a side view of a well plate having a curved bottom usable in one embodiment of the invention;

FIG. 7 is a side view of a flexible well plate having a removable top portion usable in one embodiment of the invention;

FIG. 8 is a side view of a well plate having a laminate construction usable in one embodiment of the invention;

FIG. 9 is a schematic diagram showing leveling of coating array materials by application of a leveling force and curing by hot air;

FIG. 10 is a perspective view of an example of a centrifuge usable in an embodiment of the invention;

FIG. 11 is a perspective view of an example of a swing arm centrifuge rotor assembly usable in one embodiment of the invention, showing the assembly loaded with well plates; and

FIG. 12 is a perspective view of an example of a 96-well plate usable in one embodiment of the invention.

Detailed Description of the Preferred Embodiments

In accordance with one aspect of the invention, it has been recognized that by using automation of certain development processes, miniaturization of samples to be tested, database development and manipulation, and using algorithms to identify candidate materials from information contained in databases, one can increase the number of coating materials that can be developed to meet identified needs. As used herein, the term "combinatorial" refers to the combined approach of high-throughput analysis of libraries consisting of arrays of coat material formulations. Included in the high-throughput analysis are automated or robotic processing of the sample arrays.

Combinatorial methods have been used in the medical , pharmaceutical and biotechnology industries to develop chemical compositions, particularly pharmaceuticals and medicaments, for a number of years. However, these prior combinatorial methods have not been well suited to development of new coatings. Applicants herein provide techniques for generating arrays of coating formulations, well suited to the application of combinatorial chemistry methods. These techniques allow new coatings to be screened and evaluated on a high throughput basis, in order to produce new coatings economically.

Combinatorial Approach

With reference to FIG. 1 of the drawings, which are given by way of example, and not by way of limitation, a system 10 in accordance with principles of the invention comprises a method of developing new coatings by means of a combinatorial approach. A first step 12 is to define

what end result coating is desired, and what characteristics and qualities such a coating will have. To achieve the desired result a new material, or a new construction of several materials, such as a laminate for example, comprising new and/or conventional materials combined in a novel way may be required.

At the outset it should be understood that combinatorial methods can be applied to both the process of creating coating materials by formulation or synthesis, and to creating coating parameters or desired characteristics.

Returning to consideration of one example of a combinatorial approach to coating development, the next step 13 is to select likely candidate materials. These can comprise formulations of generally dilute solutions of raw material ingredients 14 that are contemplated as likely elements or components that may provide a coating material with desired characteristics. In the next step 16 a material library of a few to a few hundred thousand, or more, chemical combinations are formed and dispensed into an array of coating wells 18 using a robot or other automated device 17 to make a library or array of coating materials . Incidentally, the "libraries" may include the samples in a single array, or the samples may form a plurality of arrays, processed either concurrently or successively. The chemical combinations forming at least part of the library are then processed in parallel as indicated at reference numeral 19. Processing can include exposing the coating array to a variety of processing variables such as heat, and time as well as applied leveling forces to shape the resultant library or array of coat samples, as can be accomplished, for example, by a centrifuge 20. In the next step 21 high throughput analysis is performed whereby the library is screened by detectors that quickly scan various properties of the coating materials. After the high throughput analysis, materials with the desired properties are identified 22 with the results entered into a large database 23, allowing up to 25,000 variations of materials to be tested at one time. Each library is comprised of one or more arrays of variations of materials to be tested. Each individual site in an array will correspond to a specific formulation of a coat material, wherein the parameter or coat descriptor(s) of the material located at that site is known. Miniaturization of the sample size facilitates processing and greatly saves cost and time thereby increasing efficiency and the rate of discovery. The end result is discovery and determination of the most successful new material(s) and the process or parameters used to produce the new materials. These materials are then selected for large scale production and commercialization 24.

The combinatorial approach to development and testing of novel coat materials greatly benefits from use of devices and apparatus that allow flat coating samples in the arrays or within

wells in the arrays. Additional embodiments encompassing such devices and apparatus are included in the present invention and further described below.

When trying to coat one formulation after another in a rapid fashion, "holdover" considerations are important. As used herein, the term "holdover" is defined as the volume of material that is residual in a cavity after it is emptied and could contaminate the next batch of material deposited into the cavity. As volumes of the cavity get smaller, the potential for holdover increases. For example, tubes, pipette tips, material dispensers and such all have potential holdover volumes. The contamination is also a function of the rheological nature or viscosity of the material that is deposited into the cavity. Holdover effects in traditional methods of developing coating materials greatly increases the level of error, compromising the identification of correct parameters of a new coat material. In the present invention, holdover and its contaminating effects are eliminated by use of a disposable dispensing device 25 (FIG. 2) and a disposable substrate assembly (formatted as a multi-well apparatus) 26, both of which are further described below. As used herein, the term "substrate" is defined as any coat-receiving surface or material, or a substance upon which a sample coat material resides which allows the testing of that sample. A "substrate assembly" is a composite of materials formed into a unit or apparatus for holding a large number of different coating samples in an array format (FIG. 3). An "array format" as used herein, is a matrix format where the samples of coating material are arranged as discrete coated areas 31 on a surface, such as a planar surface. For example, a 48-well coating array (FIG. 3) would have 48 discrete coated areas arranged as 6 rows 27 and 8 columns 28.

Multi-well Apparatus for Parallel Processing of a Material Library

An initial step in the development of a coating is to create the various mixed formulations to be placed in the wells in the array. In one embodiment of the present invention, such sample formulations can be mixed or prepared in a multi-well plate format (FIG. 3) with each individual well containing a unique, pre-defined formulation to be tested. A variety of types of commercially available multi-well plates suitable for use in the present invention can be used (Millipore Corp., Polyfiltronics, VWR Scientific). Such multi-well plates can vary in size of plate dimension, size of well (outer circumference as well as well-depth), type of material used to construct the multi-well plate (for example, polystyrene or polypropylene, rigid plastic or flexible plastic). The biotechnology and pharmaceutical industry utilizes multi-well plates (generally 48-, 96- or 256-well plates) whose outer dimensions are standardized for use with robotic dispensers. Generally, standardized multi-well plates are rectangular, rigid, stackable

plates with right edges of the top or lid portion being curved 29. The outside dimensions of a complete multi-well unit are approximately 5 x 3.25 inches. Such multi-well plates are suitable for use in the present invention. In general, the well size used should be of substantial volume so as to allow adequate robotic mixing of the required or needed amount of each formulation without drying up of the solutions contained in the wells. Preferably a well volume of .5 to 3 cubic centimeters in volume is contemplated for use in the present invention. The minimum quantity or volume of sample to be mixed in a "mother" wellplate will vary depending upon the desired coating thickness, domain size and formulation of the coating solution.

As used herein, a "mother" well plate is defined as a source well plate. For example, a 25 micron thick coating that is 1 cm^2 in domain size with a coating solution that is 50% solids, will require (1 cm^2 x 25 microns / 0.5) volume units or 0.0050 cc of solution. "Domain size" as used herein, refers to the minimum area required for the coated sample as determined by downstream testing. The appropriate volume of individual formulations from this mother well plate can then be dispensed to a sample or "daughter" well plate to make a coating with the desired domain size for subsequent analysis and data collection. It should be understood, that alternative embodiments include use of a single well plate as both the mother and daughter well plate. In such a case, the well plate into which the sample formulations are mixed will also serve as the well plate from which the coating materials will be tested. Again, considerations of desired coating thickness, domain size and formulation of coating solutions will be included in determination of minimum volume of well size required. Additional embodiments of well plate apparatus design will be discussed further below.

Automated Dispensing of Candidate Coat Materials for Testing

A disposable metering device can be used to dispense the formulations from a mother well plate to a daughter well plate. A robotic dispenser (available commercially for example, from Hamilton Zinser Packard) (FIG. 2) is one such device. Robotic dispensers allow for rapid and automated dispensing of a specified quantity of a large number of samples. The well plate format to be used for the daughter well plate will also depend on the domain size requirement of the coating. For example, a 6-, 12-, 24-, 48-, 96-, or 384- well plate format are commercially available formats which can be used in the present invention with the commercially available robotic dispensers. The robotic dispenser will have a platform area upon which the substrate well plates reside (FIG. 2; "A").

Alternatively, in the case where a single well plate is used as both the mother and daughter wellplate, a robotic device can also be used for mixing as well as dispensing component

materials for the sample coating formulation to be tested. Such a device could have multiple dispensing units 30 from which specific and precise amount of an individual component is dispensed into a single well. The sample solution can be dispensed using disposable pipette tips 30b attached to the pipettors 30c. For example, a separate dispensing unit for each component can be used to dispense the appropriate amount of a respective component into a single sample well. Such a dispensing unit can be disposable which will allow rapid and accurate automation of the combinatorial method for formulating or synthesizing a new coating with elimination of holdup or contamination problems. Examples of disposable dispensing units include, polyethylene or other type of tubing and disposable pipette tips.

Alternative Designs of Multi-well Apparatus for Parallel Processing

Alternative embodiments of well plate design include providing a two-piece coating well apparatus having at least a substrate portion 32 and a multi-well or sample-containing template 34 which can be separated from one another (FIG. 4). Once leveled and dried, the coating material 36 is held by the substrate portion 32 of the assembly. This type of well plate assembly is designed such that the base substrate-portion (or bottom half of the assembly) 32 can be removed from the multi-well template portion 34 of the well plate assembly. Various embodiments of a well plate design having a removable bottom are contemplated and further described below. FIG. 5 shows an example of a multi-well plate depicting the array format useful in the invention. Coating material samples are placed within the apertured, multi-well template top 47. Such multi-well plates will form an array 41 or library format of the different formulations as discrete coated areas 40 on a planar substrate sheet 42. A multi-well plate with a removable top or cover can also be used as a well plate assembly. An example of such a multi-well plate design is shown in FIG. 7. The well plate design can also include modifications to the well plate to prevent distribution of coating material onto the inner walls of the wells. For example, a release coating can be applied to the inner walls 43 of the wells to prevent any sample material from moving up and onto the well walls during application of a leveling force.

An additional embodiment of the present invention includes multi-well plates designed to obtain flat coatings in all of the wells of assembly. Current commercially available multi-well plates have a flat-bottom surface for the entire plate. This results in an uneven distribution of sample material in the wells located along the perimeter of the multi-well plate 68 when current swing arm type of centrifuge rotors 70 are used to apply a leveling force. FIG. 6 shows an example of a modified multi-well plate designed to obtain flat coatings in all of the wells. Such a well plate will have a curved base plate 44 where the curvature of this base is parallel to the

circumference of the centrifuge rotor, or is curved so as to substantially match the curvature of the curvilinear path of the well plate during centrifugation. With a curved-bottom well plate 44, sample material or coating solutions in all of the wells, including perimeter wells 45, will be at the same distance from the spin axis of the centrifuge. Thus, coating material in all of the wells will have a flat distribution following centrifugation. The top view of such a multi-well plate can be as depicted in FIG. 5. A flexible substrate and apertured well plate may be employed to provide a curved configuration when mounted in a centrifuge.

A specialized laminate well plate construction is also envisioned as an alternative embodiments of the present invention. FIG. 8 shows a cross sectional view of a representative laminate multi-well plate assembly. In one case, the assembly is made up of at least 4 layers and is shown in FIG.8. The top or first layer 46 corresponds to the multi-well or sample holding portion of the assembly. This layer need only be thick enough to provide a sufficient barrier between adjacent wells so that the dispensed coating material 50 does not cross contaminate adjacent samples. Where a very small amount of coating material 50 is to be tested, this layer need not be very thick and could be made of, for example, thin plastic, foam or paper with each well formed of holes placed in linear, multiple rows to form an array pattern. Preferably, the top layer will be about .01 to about 1 mm, or about1 to about 10 mm, or about 1 to about 5 cm in height. This top layer 46 can be coated with a Pressure Sensitive Adhesive (PSA) (not shown) to attach it to the substrate layer 48. This will also help to seal the wells so that cross-contamination of sample coating material from one well does not mix with its neighbors. The second layer is the substrate layer 48 and can be formed of a variety of materials, such as plastic, polymeric resin or paper, so long as it will hold the sample coating material 50 in a flattened manner. The second layer will preferably be about 1 to about 100 microns, or about 1 to about 10 mm, or about 1 to about 5 cm in thickness. The third layer is a Pressure Sensitive Adhesive layer (PSA) 52. The PSA layer 52 can be about 5 to about 30 μm , or about .005 to about .03 mm, or about.0005 to about.003 cm in thickness depending upon the type of adhesive and degree of adhesion desired. The fourth layer is a liner 54 coated with a release layer such as silicone, which can be removed or peeled away from the PSA layer 52 leaving the adhesive on the bottom of the substrate layer as the new bottom layer. This type of multi-well plate design is suitable for example, where the stickiness or tackiness of a coating material is to be tested. In such a case, it is desirable to have an array library which will remain stationary or adhere to a support surface by the PSA layer 52 while each individual coating sample is tested. Use of the PSA 52 on the layer 48 will allow the array library to remain stationary and not lift up during testing.

Leveling Force

Once the different formulations are dispensed into a multi-well plate assembly 63, the coat formulations are made into flat coatings 64 within the wells by use of a leveling force. A "leveling force" as used herein, is defined as any force sufficient to cause a sample or coat material to distribute evenly and flatly onto a substrate. A leveling force will also remove any residual air bubbles present within the sample coat formulation. A variety of leveling forces are contemplated for use in the present invention including, for example, use of centrifugal force, use of a vacuum or negative pressure force, use of an electrostatic force, or use of a magnetic force. In the case where magnetic leveling force is used, the test coat formulation will contain magnetic particles, powder, or a compound such as ferrite, that is responsive to a magnetic force. Use of a leveling force need not be limited to single-coat assessments. Where the processing of a multi-layer construction of coat material is desired, a leveling force can be repeatedly applied following dispensing of individual layers of a coat to be tested. The final array obtained will be a planar sheet containing discrete areas in a grid format of multi-layer coat formulations.

FIG. 11 shows an example of a centrifuge that can be used for applying a leveling force to a multi-well plate. Such swing arm-type centrifuges with multi-well plate holders (FIG. 12) are available commercially (for example, VWR Scientific, "MicroPlus GH 3.8 rotor centrifuge). The rotor for use in such a centrifuge is designed so as to hold an even number of multi-well plate assemblies. The multi-well plate assemblies 68 are loaded into the rotor 70 in an upright or horizontal position. During centrifugation, the plates are directed into a vertical position which then levels or flattens the sample formulations onto the substrate layer. After the formulations are dispensed in a multi-well plate assembly, the assembly is placed in a swing-arm centrifuge and the coatings are spun at controlled speeds so as to form a flat coating within each well 64. For example, with a standard centrifuge, a 10-min. spin at 2000 rpm will be sufficient to evenly distribute the coat materials within each well. There is no loss of sample material with use of a swing-arm centrifuge.

Additional methods of casting sample coat formulations include those which can also simultaneously dry the coating material during casting. For example, a centrifuge which has been modified to hold circulating hot air or other gas which will aid in the evaporation of carrier solvents in the coating formulations is also contemplated for use in the present invention and is diagrammed schematically in FIG. 9. The hot air 66 circulating over the formulations during centrifugation aids in the drying of the coating by evaporation of volatiles or solvents. As with a centrifuge, devices used to provide alternative methods of applying a leveling force can also be modified so as to simultaneously dry the coat formulations. For example, an apparatus utilizing

a vacuum or electrostatic force as the leveling force can be modified to circulate hot air and include alternate arrangements for drying.

High Throughput Analysis, Data Storage, Data Modeling and New Materials Discovery

The above methods provide an array 40 of coating materials with each site in the grid array containing a coat material having a known parameter which differs from parameter values of the materials contained on the other sites (FIG 1; step 16). With this array, the plurality of coating materials can each be tested for performance of each coating. Because the parameter value of the coating contained at each site is known, the value of a parameter associated with a desired performance of a coating can be determined. All information obtained by this high throughput analysis screening a coat material library are then entered into a database. From this database identification of the most successful new coat materials and the parameters and descriptors used to produce them is achieved (FIG. 1, step 23). Such a database will also serve as a storage library to aid in the formulation of future parameters to characterize the coatings.

Example I

This example demonstrates the use of a multi-well plate combined with a centrifugal leveling force for estimation of coat weight of a sample coat material formulation. This example is intended to be representative of one embodiment of the invention, and not intended as limiting the scope of the invention.

The emulsion polymer formulation used was S-2000. S-2000 is a nondispersable emulsion acrylic polymer manufactured by Avery Dennison Corporation, Pasadena CA in accordance with U.S. Patent No. 5,221,706. A 96-well plate obtained from Polytronics was used as a daughter well plate. The well plate remained flat during centrifugation. Each well contained an equivalent sample material formulation for determination of coat weight.

Diameter of each well = 0.6 cm

Cross-section of each well = 3.14 x 0.6 cm2 = 1.884 cm2

Weight of coat material in E7 position of array = 0.0153 gm

Wet coat weight in E7 = 0.0153/.0001884 = 81.21 gsm

% solids in wet solution = 52.1%

Dry coat weight in E7 = 42.3 gsm

Results:

The emulsion did not dry fast and remained opaque. Hence the need for higher temperature drying. Material in wells located on the perimeter wells did not level evenly. Coat material dispensed into the center wells were centered and evenly flattened in the horizontal direction. The uneven leveling observed in the perimeter wells is believed to be a result of the centrifugal force acting at an angle to the bottom of the well, unlike the preferred flexible configuration of Fig. 6.

This example demonstrates the utility of using a multi-well plate combined with a leveling force for high-throughput analysis of specific parameters or characteristics of coat material formulations in an individualized manner.

The invention has been described with reference to various specific and preferred embodiments and techniques. However, it should be understood that many variations and modifications may be made while remaining within the spirit and scope of the invention

CLAIMS

1. A method for efficiently preparing a large number of sample coatings comprising the steps of:

(a) forming a series of sample receptacles or wells by providing a flexible substrate and an overlying apertured sheet with the apertured sheet in tight sealing engagement with the substrate;

(b) applying different samples of material in liquid form into said receptacles;

(c) placing said flexible substrates with said sample receptacles thereon in a centrifuge;

(d) activating said centrifuge with said receptacle mounted therein to flatten out the sample material in said receptacles, with the centrifugal force acting perpendicular to the bottom of the receptacles;

(e) drying said samples while they are within the centrifuge; and

(f) removing the apertured plate to leave the samples exposed on said substrate.

2. A method as defined in claim 1 wherein said applying step involves the application of various adhesive compositions into said receptacles or wells.

3. A method as defined in claim 1 wherein multilayer samples are formed by repeating steps (b) through (e) prior to step (f).

4. A method as defined in claim 1 wherein an array of at least four wells are formed.

5. A method as defined in claim 1 wherein hot air is applied to the samples during centrifugation.

6. A method as defined in claim 1 wherein said substrate is formed of paper.

7. A method of forming a test coating comprising the steps of:

forming a receptacle for receiving a material sample, said receptacle having a flat bottom and enclosing sides:

depositing a fluid material sample in said receptacle;

mounting said receptacle in a centrifuge with the outward centrifugal force being perpendicular to the bottom of said receptacle;

activating said centrifuge to flatten the material in the receptacle; and

drying said material while the sample is being rotated and flattened by the centrifugal action.

8. A method for efficiently preparing a large number of sample castings comprising the steps of:

forming a series of sample receptacles by providing a substrate and an overlying apertured sheet with the apertured sheet in tight sealing engagement with the substrate;

applying different samples of material in liquid form into said receptacles;

drying said samples; and

removing said apertured sheet to leave said material samples on said substrate.

9. A method as defined in claim 8 including the step of applying force to said samples perpendicular to the bottom of said receptacles to flatten out said samples.

10. A method of testing coating materials, comprising the steps of:

providing an array of coating wells, each well being configured for receiving a coating material having a known composition;

placing a coating material having a known composition in each coating well, varying the composition so as to provide a plurality of coating materials having different compositions in a plurality of coating wells;

correlating the composition of the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a specific composition is associated with each coating well position in the array;

placing said coating wells with said compositions into a centrifuge, and activating said centrifuge;

drying said coating materials; and

testing the resultant coatings.

11. The method of claim 10 including providing wells in the form of a flexible substitute and a flexible overlying apertured sheet.

12. method of claim 10 including the step of heating said coating materials while said centrifuge is activated.

13. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter; said array of coating wells comprising a substrate and an overlying apertured sheet;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

drying said coating samples; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

14. The method of claim 13, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after drying, whereby the coating material array is carried by the substrate alone after separation.

15. The method of claim 13, wherein the well depth and volume is substantially greater than that of the coating volume.

16. A method of analyzing coating materials for performance of the coating with regard to a property of a coating, comprising:

providing an array of coating wells, each well being configured for receiving a coating material having a known parameter;

placing a coating material having the known parameter in each coating well, varying the parameter so as to provide a plurality of coating materials having different parameter values in a plurality of coating wells;

correlating the value of the parameter for the coatings deposited in each of the plurality of coating wells with the position of the coating well in the array, whereby a parameter value is associated with each coating well position in the array;

applying a centrifugal force to the array of coating wells to level the coating material in the coating wells;

curing said coating samples under said coating leveling force; and

testing the coatings in the array to analyze the relationship between the position in the array and performance with regard to the property of the coating material;

whereby the value of the parameter can be correlated to the performance of the coating with regard to the property of the coating.

17. The method of claim 16, further comprising the steps of:

providing a coating well apparatus having at least a substrate part and a well wall part which can be separated;

separating the well wall part from the substrate part after application of the leveling force, whereby the coating material array is carried by the substrate alone after separation.

18. The method of claim 10, further comprising the steps of:

curving the said array of coating wells to substantially match the curvature of the curvilinear path of the array during centrifuging.

ABSTRACT

A combinatorial, high-throughput screening method is described for developing new coatings having a desired performance characteristic of a coating property which results in a substantial increase in the discovery rate of new coating materials. The method includes the steps of providing an array of wells (18) for receiving candidate coating materials have a known parameter, placing coating materials in each well (16) while varying the coating material parameter, correlating the coating material position in the array to the variation of the coating material parameter; applying a coating leveling force to and optionally drying the coating materials in the array of coating wells (19); testing the coatings with regard to the desired performance characteristic (21) and correlating the result of the test to the well position in the array that thereby coating materials having the desired performance characteristic may be discovered.